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ore Patent Application of:

Craig N. Eatough, et al.

Docket:

8333.Appeal

Serial No.:

09/954,603

Art Unit:

1764

DEC 0 1 2003

Filed:

September 17, 2001

Examiner: Alexa A. Doroshenk

CLEAN PRODUCTION OF COKE

APPENDIX ON APPEAL

Mail Stop Appeal Briefs - Patents Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

Sir:

I hereby certify that this correspondence is being deposited with the United States Postal Service as first class mail in an envelope addressed to: Mail Stop Appeal Briefs - Patents, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450 on November 26, 2003.

TABLE OF CONTENTS - APPENDIX

| Appealed Claims 32-69 Al |
|-------------------------------------|
| Advisory Action Mailed 10/16/03 |
| Office Action Mailed 08/25/03 |
| Office Action Mailed 12/26/02 |
| Declaration of L. Douglas Smoot |
| Weber U.S. Pat. 4,352,720 |
| Deering U.S. Pat. 4,530,752 |
| Nicaud U.S. Pat. 6,043,289 |
| Applicants' Amendment faxed 9/19/03 |

- - 32. A method of producing high grade coke from low grade material without causing a pollution problem, comprising the acts of:

introducing a mixture of unwashed non-elutriated low grade non-coking inexpensive coal fines and another type of inexpensive carbonaceous fines comprised of unwashed non-elutriated waste coke fine, as a feedstock influent into a pyrolyzer;

pyrolyzing the mixture in the pyrolyzer;

discharging coke and pyrolytic by-products as effluents from the pyrolyzer. - -

- - 33. A method according to Claim 32 further comprising the acts of:

feeding back tar effluent by-product from the pyrolyzer to the feedstock influent mixture; feeding back combustible off gas effluent by-product from the pyrolyzer to the pyrolyzer and using it as a source of fuel in the pyrolyzer. - -

- - 34. A method according to Claim 32 further comprising the act of obtaining a mixture comprising unwashed waste coal fines and unwashed waste coke fines prior to the introducing act. --
- - 35. A method according to Claim 32 further comprising the act of crushing low grade coal and/or the carbonaceous waste coke prior to the introducing act, to obtain the fines. -
- - 36. A method according to Claim 32 further comprising the act of forming the mixture into solid objects prior to the introducing act. -
- - 37. A method according to Claim 35 wherein the discharging act comprises discharging the coke as solid objects. -

- - 38. A method according to Claim 33 wherein the first feeding act comprises combining the feedback tar, a synthetic binder and the mixture of fines prior to the introducing act. -
- - 39. A method according to Claim 33 wherein the by-product tar is fed back mixed with another binder additive and combined with the mixture of coal fines and waste coke fines prior to the introducing act. -
- - 40. A method according to Claim 32 wherein the discharging act comprises cooling the by-products and condensing tar to separate the tar from off gas. -
- - 41. A method of producing coke from a mixture of non-prime coal fines and waste coke fines comprising the acts of:

introducing a mixture of non-elutriated low grade coal fines and another type of carbonaceous comprising non-elutriated waste coke fines as a feedstock influent into a pyrolyzer;

pyrolyzing the mixture in the pyrolyzer;

discharging segregated coke and pyrolytic by-products as effluents from the pyrolyzer. - -

-- 42. A method according to Claim 41 further comprising the acts of:

separating the pyrolytic by-products into tar and combustible off gas;

combining the separated tar as a binder with the mixture of coal and coke fines in the mixture;

returning the combustible off gas to the pyrolyzer as a source of fuel. - -

- - 43. A method according to Claim 41 wherein the introducing act comprises obtaining a mixture comprising non-elutriated waste coke fines and non-elutriated waste coal fines. -
- -- 44. A method according to Claim 41 further comprising the act of crushing at least some of the coke and/or the coal, prior to the introducing act. --
- -- 45. A method according to Claim 41 further comprising the act of forming the mixture into solid objects prior to the introducing act. --
- -- 46. A method according to Claim 45 wherein the discharging act comprises discharging the coke from the pyrolyzer as solid objects. --
- - 47. A method according to Claim 42 wherein the combining act comprises combining the separated tar, a synthetic binder and the mixture of coal and coke fines prior to the introducing act. -
- -- 48. A method according to Claim 42 wherein the separated tar is fed back to the coal and coke mixture prior to the introducing act. --
- - 49. A method according to Claim 42 wherein the separating act comprises cooling the by-products to condense tar to separate the tar from off gas. -

-- 50. A method of producing coke from low grade coal and coke fines, comprising the acts of:

obtaining and mixing unwashed low grade coal fines and unwashed coke fines;

introducing the unwashed mixture of lower grade coal fines and waste coke fines as an influent into a pyrolyzer;

pyrolyzing the unwashed mixture in the pyrolyzer;

discharging segregated coke, on the one hand, and pyrolytic by-products comprising combustible off gas and tar on the other hand, as effluents from the pyrolyzer;

separating the pyrolytic by-products into segregated tar and combustible off-gas; adding the segregated tar as a binder to the coal and coke fines mixture; returning the segregated combustible off gas to the pyrolyzer as a source of fuel. --

- -- 51. A method according to Claim 50 further comprising the act of crushing oversized unwashed waste coke and/or unwashed oversized low grade coal, to correctly size the fines. --
- - 52. A method according to Claim 50 further comprising the act of forming the mixture into solid objects to the introducing act. -
- -- 53. A method according to Claim 52 wherein the discharging act comprises discharging the coke from the pyrolyzer as solid objects. --
- - 54. A method according toe Claim 50 wherein the adding act comprises combining the separated tar, a synthetic binder and the mixture of coal and coke fines prior to the introducing act. --

- --55. A method according to Claim 50 wherein the separated tar is fed back to the mixture of coal and coke fines. --
- -- 56. A method according to Claim 50 wherein low grade coal comprises 20-40% by weight of the coal and coke mixture. --
- -- 57. A method according to Claim 50 wherein the coke fines comprise petroleum coke fines which comprise 40-70% by weight of the coal and coke mixture. --
- -- 58. A method according to Claim 50 wherein the coke fines comprise coke breeze fines which comprise 5-10% by weight of the coal and coke mixture.
- -- 59. A method according to Claim 50 wherein the pyrolyzing act comprises heating the introduced mixture to a temperature within the range of 800-1100°C at a rate within the range of 1500-2000°C/hour to lower coke volatility below 2%. --
- -- 60. A method according to Claim 50 wherein the separating act comprises cooling the by-products to about 300°C and condensing the tar to separate the tar from the off gas. --

-- 61. A method of producing high quality coke from a mixture of low grade and/or waste carbonaceous materials at a much lower cost comprising the acts of:

absent elutriation, introducing a mixture of low grade coal fines and waste coke fines as an influent into a pyrolyzer;

pyrolyzing the mixture of fines in the pyrolyzer;

discharging the coke, and pyrolytic by-products from the pyrolyzer. - -

-- 62. A method according to Claim 61 wherein the by-products comprise tar and combustible gas and further comprising the acts of:

condensing the tar;

using the tar as a binder for the mixture of coal and coke;

using the combustible off-gas as a source of fuel in the pyrolyzer. --

-- 63. A method of producing coke from non-traditional carbonaceous materials comprising the acts of:

introducing a mixture of unwashed waste coke fines and unwashed non-coking grade coal fines as an influent into a pyrolyzer;

pyrolyzing the mixture in the pyrolyzer;

discharging the coke, and pyrolytic by-products comprising combustible off-gas and tar as effluents from the pyrolyzer. - -

- -- 64. A method according to Claim 63 comprising the further acts of: condensing the tar to separate the tar and off-gas; using the tar as a binder for the mixture fines prior to the mixing act; using the combustible off-gas as a source of fuel in the pyrolyzer. --
- -- 65. A method according to Claim 64 wherein all condensed tar is utilized as binder and all combustible off-gas is used to fuel the pyrolyzer. --
- -- 66. A method according to Claim 64 wherein the condensed tar is the sole binder source and the combustible off-gas is the sole source of fuel for the pyrolyzer. --
- - 67. A method of cost effectively producing high quality coke from a mixture of non-traditional carbonaceous materials comprising the acts of:

without washing or elutriation, introducing into a pyrolyzer a mixture comprising low grade coal fines and coke fines as salvage from prior production of coke;

pyrolyzing the mixture and obtaining segregated coke and by-products. - -

-- 68. A method of producing coke, comprising the acts of:

mixing a binder, low grade non-prime unwashed coal fines selected from the group consisting of unwashed waste non-coking coal fines and unwashed non-coking coal fines and unwashed salvage coke fines selected from the group consisting of unwashed waste petroleum fines, unwashed waste char fines and unwashed waste coke breeze, without regard to a free swelling index value;

introducing the mixture into a pyrolyzer;

pyrolyzing the mixture to derive coke, tar and combustible off-gas. --

-- 69. A method according to Claim 68 wherein the method is performed in a closed system and further comprising the acts of:

causing all of the tar to comprise the binder;

fueling the pyrolyzer with the combustible off-gas. - -



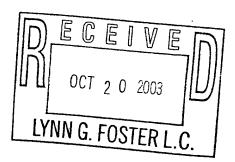
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| APPLICATION NO. | FILING DATE | FIRST NAMED INVENTOR | ATTORNEY DOCKET NO. | CONFIRMATION NO. | |
|---|---------------|----------------------|---------------------|------------------|--|
| 09/954,603 | 09/17/2001 | Craig N. Eatough | 8333 8272 | | |
| 75 | 90 10/16/2003 | | EXAM | EXAMINER | |
| Foster & Foster, LLC | | | DOROSHENK, ALEXA A | | |
| Mr. Lynn G. Foster 602 E. 300 S. Salt Lake City, UT 84102 | | | ART UNIT | PAPER NUMBER | |
| | | | 1764 | | |

DATE MAILED: 10/16/2003

Please find below and/or attached an Office communication concerning this application or proceeding.



| ŗ | • | Application No. | Applicant(s) | | | | |
|------|--|----------------------------------|--------------------------------------|--|--|--|--|
| | Advisory Action | 09/954,603 | EATOUGH ET AL. | | | | |
| | | Examiner | Art Unit | | | | |
| 7 | | Alexa A. Doroshenk 🕬 | 1764 | | | | |
| | The MAILING DATE of this communication app | ars on th cov r sheet with the c | correspondence address | | | | |
| | THE REPLY FILED 9-19-03 FAILS TO PLACE THIS APPLICATION IN CONDITION FOR ALLOWANCE. Therefore, further action by the applicant is required to avoid abandonment of this application. A proper reply to a final rejection under 37 CFR 1.113 may only be either: (1) a timely filed amendment which places the application in condition for allowance; (2) a timely filed Notice of Appeal (with appeal fee); or (3) a timely filed Request for Continued Examination (RCE) in compliance with 37 CFR 1.114. | | | | | | |
| | | PLY [check either a) or b)] | | | | | |
| | a) The period for reply expiresmonths from the mailing date of the final rejection. The period for reply expires on: (1) the mailing date of this Advisory Action, or (2) the date set forth in the final rejection, whichever is later. In no event, however, will the statutory period for reply expire later than SIX MONTHS from the mailing date of the final rejection. ONLY CHECK THIS BOX WHEN THE FIRST REPLY WAS FILED WITHIN TWO MONTHS OF THE FINAL REJECTION. See MPEP 706.07(f). Extensions of time may be obtained under 37 CFR 1.136(a). The date on which the petition under 37 CFR 1.136(a) and the appropriate extension fee have been filed is the date for purposes of determining the period of extension and the corresponding amount of the fee. The appropriate extension fee under 37 CFR 1.17(a) is calculated from: (1) the expiration date of the shortened statutory period for reply originally set in the final Office action; or (2) as set forth in the bearing date of the final rejection, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b). | | | | | | |
| | 1. A Notice of Appeal was filed on Appellant's Brief must be filed within the period set forth in 37 CFR 1.192(a), or any extension thereof (37 CFR 1.191(d)), to avoid dismissal of the appeal. 2. The proposed amendment(s) will not be entered because: | | | | | | |
| | | | | | | | |
| | (a) \(\sum_{\text{they raise new issues that would require further consideration and/or search (see NOTE below);} \) (b) \(\sum_{\text{they raise the issue of new matter (see Note below);} \) | | | | | | |
| | (c) they are not deemed to place the application in | | afalls and the second | | | | |
| | losacs for appeal, allu/of | | | | | | |
| | (d) they present additional claims without canceling | g a corresponding number of fir | nally rejected claims. | | | | |
| | NOTE: <u>See Continuation Sheet</u> . | | | | | | |
| | 3. Applicant's reply has overcome the following rejection | on(s): | | | | | |
| | 4. Newly proposed or amended claim(s) would be allowable if submitted in a separate, timely filed amendment canceling the non-allowable claim(s). | | | | | | |
| , | 5.⊠ The a)⊠ affidavit, b)□ exhibit, or c)□ request for reconsideration has been considered but does NOT place the application in condition for allowance because: <u>See Continuation Sheet.</u> | | | | | | |
| • | 6. The affidavit or exhibit will NOT be considered because it is not directed SOLELY to issues which were newly raised by the Examiner in the final rejection. | | | | | | |
| 7 | 7. For purposes of Appeal, the proposed amendment(s) a) will not be entered or b) will be entered and an explanation of how the new or amended claims would be rejected is provided below or appended. | | | | | | |
| | The status of the claim(s) is (or will be) as follows: | | | | | | |
| | Claim(s) allowed: | | | | | | |
| | Claim(s) objected to: | | | | | | |
| | Claim(s) rejected: | | | | | | |
| | Claim(s) withdrawn from consideration: | | | | | | |
| | . The proposed drawing correction filed on is a)[| ☐ approved or b)☐ disappro | ved by the Examiner | | | | |
| 9 | . Note the attached Information Disclosure Statement(s | s)(PTO-1449) Paper No(s). | | | | | |
| 10 | O. Other: | (| Teny Down | | | | |
| S. P | atent and Trademark Office | | JERRY D. JOHNSON PRIMARY EXAMINER | | | | |
| TO | L-303 (Rev. 04-01) | | | | | | |

U.S. Patent and Trademark Offic PTOL-303 (Rev. 04-01)

Continuation of 2. NOTE: The proposed amendments raise new issues (such as 112 second paragraph) and would require further consideration.

Continuation of 5. does NOT place the application in condition for allowance because: the affidavit is not persuasive in that Smoot, who is a part owner/assignee of the application, is giving a legal opinion on the sufficiency of the disclosure. From paragraph 6 of the declaration: "I conclude, as one having skill in this art, that there is no disclosure of washing or elutriating of the fines being displaced using the equipment and methodology disclosed to produce coke. Thus, in my opinion, it is correct to say the limitations of "unwashed" and "non-elutriated" in the claims are properly supported by the present specification because the fines displacement are neither washed nor elutriated."

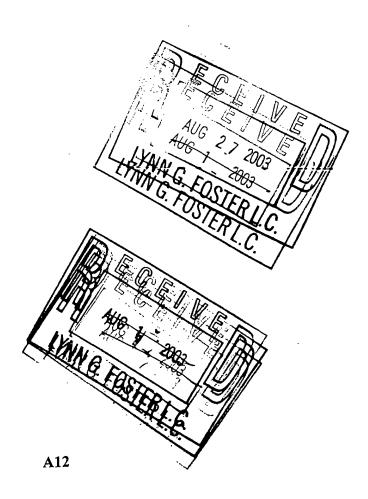


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| 09/954,603 09/17/2001 | | Craig N. Eatough | 8333 | 8272 |
| 75 | 90 08/25/2003 | | | |
| Foster & Foster, LLC Mr. Lynn G. Foster 602 E. 300 S. | | EXAMINER | | |
| | | | DOROSHENK, ALEXA A | |
| Salt Lake City, | UT 84102 | | ART UNIT | PAPER NUMBER |
| | | | 1764 | |
| | | | DATE MAILED: 08/25/2003 | |

Please find below and/or attached an Office communication concerning this application or proceeding.



PTO-90C (Rev. 07-01)

| · | | | | | | |
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| | | Application No. | Applicant(s) | | | |
| Office Action S | Office And on O | 09/954,603 | EATOUGH ET AL. | | | |
| Office Action St | ımmary | Examiner | Art Unit | | | |
| | | Alexa A. Doroshenk | 1764 | | | |
| The MAILING DATE of Period for Reply | this communication ap | ppears on the cover sheet with th | he correspondence address | | | |
| THE MAILING DATE OF THI - Extensions of time may be available un after SIX (6) MONTHS from the mailing - If the period for reply specified above is - If NO period for reply is specified above - Failure to reply within the set or extend | S COMMUNICATION neer the provisions of 37 CFR 1 g date of this communication. It is stant thirty (30) days, a ree, the maximum statutory period ed period for reply will, by statution three months after the mailinant three months after the maximum three months after the maxi | 1.136(a). In no event, however, may a reply beply within the statutory minimum of thirty (30) | pe timely filed) days will be considered timely. from the mailing date of this communication. ONED (35 U.S.C. & 133). | | | |
| 1)⊠ Responsive to commu | nication(s) filed on <u>05</u> | June 2003 . | | | | |
| 2a) This action is FINAL . | 2b)□ T | his action is non-final. | | | | |
| 3) Since this application is closed in accordance via Disposition of Claims | s in condition for allow vith the practice under | vance except for formal matters, r <i>Ex parte Quayle</i> , 1935 C.D. 11 | , prosecution as to the merits is 1, 453 O.G. 213. | | | |
| 4)⊠ Claim(s) <u>32-69</u> is/are pe | ending in the applicati | on. | | | | |
| 4a) Of the above claim(s | ;) is/are withdra | wn from consideration. | | | | |
| 5) Claim(s) is/are al | lowed. | | | | | |
| 6)⊠ Claim(s) <u>32-69</u> is/are rej | ected. | | | | | |
| 7) Claim(s) is/are ob | ected to. | | | | | |
| 8) Claim(s) are subject Application Papers | ect to restriction and/c | or election requirement. | | | | |
| 9)☐ The specification is objec | 9)☐ The specification is objected to by the Examiner. | | | | | |
| 10)☐ The drawing(s) filed on _ | is/are: a) 🗌 acce | pted or b) objected to by the Ex | kaminer. | | | |
| | | e drawing(s) be held in abeyance. | | | | |
| | 11) The proposed drawing correction filed on is: a) approved b) disapproved by the Examiner. | | | | | |
| If approved, corrected drawings are required in reply to this Office action. | | | | | | |
| 12) The oath or declaration is | objected to by the Ex | caminer. | | | | |
| Priority under 35 U.S.C. §§ 119 a | nd 120 | | | | | |
| 13) Acknowledgment is made | e of a claim for foreigr | n priority under 35 U.S.C. § 119 | (a)-(d) or (f). | | | |
| a) | | | | | | |
| 1. Certified copies of | the priority document | s have been received. | | | | |
| <u></u> | | s have been received in Applica | ation No | | | |
| Copies of the certification from | fied copies of the prior in the International Bui | rity documents have been receive reau (PCT Rule 17.2(a)). of the certified copies not receive. | ved in this National Stage | | | |
| | | | e) (to a provisional application). | | | |
| | e foreign language pro | ovisional application has been re | eceived. | | | |
| Attachment(s) | | · · · | | | | |
| Notice of References Cited (PTO-892) Notice of Draftsperson's Patent Drawi) Information Disclosure Statement(s) (| ing Review (PTO-948) | 5) Notice of Informa | ary (PTO-413) Paper No(s) Il Patent Application (PTO-152) | | | |

A13

Art Unit: 1764

DETAILED ACTION

Claim Rejections - 35 USC § 112

1. The following is a quotation of the first paragraph of 35 U.S.C. 112:

The specification shall contain a written description of the invention, and of the manner and process of making and using it, in such full, clear, concise, and exact terms as to enable any person skilled in the art to which it pertains, or with which it is most nearly connected, to make and use the same and shall set forth the best mode contemplated by the inventor of carrying out his invention.

2. Claims 32-69 are rejected under 35 U.S.C. 112, first paragraph, as failing to comply with the written description requirement. The claim(s) contains subject matter which was not described in the specification in such a way as to reasonably convey to one skilled in the relevant art that the inventor(s), at the time the application was filed, had possession of the claimed invention. The subject matter not described in the application as filed is that directed toward "unwashed" and/or "non-elutriated" coal or coke fines. Applicant has added this subject matter to all independent claims (32, 41, 50, 61, 63, 67 and 68) by amendment. Since such subject matter was not described or conveyed in the application as filed, doubt is raised as to possession of the claimed invention at the time of filing.

The examiner makes note that if applicant were to delete the above identified new matter from the claims, the rejection of claims as presented in Paper No. 5 would continue to apply.

3. The following is a quotation of the second paragraph of 35 U.S.C. 112:

The specification shall conclude with one or more claims particularly pointing out and distinctly claiming the subject matter which the applicant regards as his invention.

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Art Unit: 1764

4. Claim 50 is rejected under 35 U.S.C. 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention.

Claim 50 is unclear as to what applicant intends to claim by the phrase "on the one hand" and "on the other hand".

Response to Arguments

35 USC 112, Second Paragraph

The examiner makes note that applicant did not present any response with regard to the 35 USC 112, second paragraph rejections. The examiner could hold their response as non-response under such conditions, but has elected not to in order to prevent further delay of the prosecution of this application.

The 35 USC 112, second paragraph rejection of claim 50, previously claim 17, has been maintained.

35 USC 102 and 103 Rejections

Applicant argues that the primary reference, Weber, does not disclose wherein the process does not use unwashed and/or non-elutriated low grade coal and coke fines.

While the examiner does agree that Weber does not disclose such, a 35 USC 112, first paragraph rejection with regard to this specific subject matter has been applied above and there for argument is considered moot.

In response to applicant's argument that the examiner's conclusion of obviousness is based upon improper hindsight reasoning, it must be recognized that

Application/Control Number: 09/954,603

Art Unit: 1764

any judgment on obviousness is in a sense necessarily a reconstruction based upon hindsight reasoning. But so long as it takes into account only knowledge which was within the level of ordinary skill at the time the claimed invention was made, and does not include knowledge gleaned only from the applicant's disclosure, such a reconstruction is proper. See *In re McLaughlin*, 443 F.2d 1392, 170 USPQ 209 (CCPA 1971).

Page 4

In response to applicant's argument that the prior art does not solve the "problems" recognized by applicant, the fact that applicant has recognized another advantage which would flow naturally from following the suggestion of the prior art cannot be the basis for patentability when the differences would otherwise be obvious. See *Ex parte Obiaya*, 227 USPQ 58, 60 (Bd. Pat. App. & Inter. 1985).

Conclusion

5. Applicant's amendment necessitated the new ground(s) of rejection presented in this Office action. Accordingly, **THIS ACTION IS MADE FINAL**. See MPEP § 706.07(a). Applicant is reminded of the extension of time policy as set forth in 37 CFR 1.136(a).

A shortened statutory period for reply to this final action is set to expire THREE MONTHS from the mailing date of this action. In the event a first reply is filed within TWO MONTHS of the mailing date of this final action and the advisory action is not mailed until after the end of the THREE-MONTH shortened statutory period, then the shortened statutory period will expire on the date the advisory action is mailed, and any extension fee pursuant to 37 CFR 1.136(a) will be calculated from the mailing date of

Art Unit: 1764

the advisory action. In no event, however, will the statutory period for reply expire later than SIX MONTHS from the date of this final action.

6. Any inquiry concerning this communication or earlier communications from the examiner should be directed to Alexa A. Doroshenk whose telephone number is 703-305-0074. The examiner can normally be reached on Monday - Thursday from 9:00 AM - 7:30 PM.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Glenn Caldarola can be reached on 703-308-6824. The fax phone number for the organization where this application or proceeding is assigned is (703) 872-9306.

Any inquiry of a general nature or relating to the status of this application or proceeding should be directed to the receptionist whose telephone number is 703-308-0661.

WAD Wante

August 13, 2003

JERRY D. JOHNSON PRIMARY EXAMINER GROUP 1100



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| APPLICATION NO. | FILING DATE | FIRST NAMED INVENTOR | ATTORNEY DOCKET NO. | CONFIRMATION NO |
|---|---------------|----------------------|-------------------------|-----------------|
| 09/954,603 | 09/17/2001 | Craig N. Eatough | 8333 | 8272 |
| 759 | 00 12/26/2002 | | | |
| Foster & Foster, LLC Mr. Lynn G. Foster 602 E. 300 S. | | | EXAMINER | |
| | | | DOROSHENK, ALEXA A | |
| Salt Lake City, U | JT 84102 | | ART UNIT | PAPER NUMBER |
| | | | 1764 | |
| | | | DATE MAILED: 12/26/2002 | \ |

Please find below and/or attached an Office communication concerning this application or proceeding.



| | Application No. | Applicant(s) | MK- |
|--|--|--|------------|
| | 09/954,603 | EATOUGH ET AL. | |
| Office Action Summary | Examiner | Art Unit | |
| | Alexa A. Doroshenk | 1764 | |
| The MAILING DATE of this commu | nication app ars on the cover sheet with | | ss |
| Period for Reply A SHORTENED STATUTORY PERIOD I THE MAILING DATE OF THIS COMMUN - Extensions of time may be available under the provision after SIX (6) MONTHS from the mailing date of this com - If the period for reply specified above is less than thirty (- If NO period for reply is specified above, the maximum s | IICATION. Is of 37 CFR 1.136(a). In no event, however, may a reply imunication. (30) days, a reply within the statutory minimum of thirty (30) tatutory period will apply and will expire SIX (6) MONTHS | be timely filed D) days will be considered timely. | unication. |
| Failure to reply within the set or extended period for repl Any reply received by the Office later than three months earned patent term adjustment. See 37 CFR 1.704(b). Status | y will, by statute, cause the application to become ABANE after the mailing date of this communication, even if timel | DONED (35 U.S.C. § 133). y filed, may reduce any | |
| 1) Responsive to communication(s) fi | iled on 13 February 2002 | | |
| | 2b)⊠ This action is non-final. | | |
| | n for allowance except for formal matters | o procesuition on to the | |
| closed in accordance with the prac Disposition of Claims | tice under <i>Ex parte Quayle</i> , 1935 C.D. 1 | 1, 453 O.G. 213. | ens is |
| 4) Claim(s) $\underline{1-31}$ is/are pending in the | application. | | |
| 4a) Of the above claim(s) is/a | re withdrawn from consideration. | | |
| 5) Claim(s) is/are allowed. | | | |
| 6)⊠ Claim(s) <u>1-31</u> is/are rejected. | | | |
| 7) Claim(s) is/are objected to. | | | |
| 8) Claim(s) are subject to restric | ction and/or election requirement. | | |
| Application Papers | · | | |
| 9)☐ The specification is objected to by the | | | |
| 10) The drawing(s) filed on is/are: | a) accepted or b) objected to by the E | xaminer. | |
| Applicant may not request that any obje | ection to the drawing(s) be held in abeyance. | See 37 CFR 1.85(a). | |
| 11)☐ The proposed drawing correction filed | l on is: a)∏ approved b)∏ disap | proved by the Examiner. | |
| If approved, corrected drawings are req | | | |
| 12)☐ The oath or declaration is objected to | by the Examiner. | | |
| Priority under 35 U.S.C. §§ 119 and 120 | | | |
| 13) Acknowledgment is made of a claim | for foreign priority under 35 U.S.C. § 119 | a)-(d) or (f). | |
| a) ☐ All b) ☐ Some * c) ☐ None of: | | · · · · · · · · · · · · · · · · · · · | |
| 1. Certified copies of the priority of | documents have been received. | | |
| _ | documents have been received in Applica | ation No. | |
| 3. Copies of the certified copies of | f the priority documents have been recei | ived in this National Stage | • |
| | | | |
| 14) Acknowledgment is made of a claim for | usage provisional application has been | d(e) (to a provisional applic | cation). |
| a) The translation of the foreign lang | r domestic priority under 35 U.S.C. && 13 | eceived. 20 and/or 121 | |
| ttachment(s) | 12 | CO GITG/OF 12 [. | |
| Notice of References Cited (PTO-892) Notice of Draftsperson's Patent Drawing Review (PTO) Information Disclosure Statement(s) (PTO-1449) Pap | O-948) 5) Notice of Informa | ary (PTO-413) Paper No(s) Il Patent Application (PTO-152) | |
| Patent and Trademark Office O-326 (Rev. 04-01) | Office Action Summary | Part of Paper | N= 5 |

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DETAILED ACTION

Claim Rejections - 35 USC § 112

- 1. The following is a quotation of the second paragraph of 35 U.S.C. 112:
 - The specification shall conclude with one or more claims particularly pointing out and distinctly claiming the subject matter which the applicant regards as his invention.
- 2. Claims 17 and 26 are rejected under 35 U.S.C. 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention.

Claim 17 is unclear as to what applicant intends to claim by the phrase "on the one hand" and on the other hand". For examination purposes, the examiner has treated these phrases to mean discharging both coke and by-products as effluents from the pyrolyzer.

Claim 26 is unclear as to what "heating the introduced mixture to a temperature within the range of 800-1100°C at a rate within the range of 1500-2000°C/hour" means. For examination purposes, the examiner, as can best be understood, has treated the claim as meaning heating the introduced mixture from 800-1100°C.

Claim Rejections - 35 USC § 102

3. The following is a quotation of the appropriate paragraphs of 35 U.S.C. 102 that form the basis for the rejections under this section made in this Office action:

A person shall be entitled to a patent unless -

⁽b) the invention was patented or described in a printed publication in this or a foreign country or in public use or on sale in this country, more than one year prior to the date of application for patent in the United States.

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4. Claims 1, 3-5, 7-9, 11-13, 15, 16, 28 and 29 are rejected under 35 U.S.C. 102(b) as being anticipated by Weber et al. (4,352,720).

With respect to claims 1, 9, 28 and 29, Weber et al. discloses a method of producing coke comprising the steps of:

introducing a mixture of coal fines into a pyrolyzer (coking oven) (col. 2, lines 10-19);

pyrolyzing the mixture (col. 2,lines 15-17);

discharging coke (45) and by-products from the pyrolyzer (69);

separating they pyrolytic by-products by condensing means (fig. 2) into tar (31) and off gas (78);

using the tar as a binder in the mixture without discharging to the environment (col. 2,lines 43-46 and col. 7, lines 44-48); and

using the off gas as a source of fuel in the pyrolyzer without discharging to the environment (col. 7, lines 25-30).

With respect to claims 3, 11 and 18, Weber et al. discloses wherein coal is crushed prior to introducing (col. 6, lines 1-4).

With respect to claims 4, 12 and 19, Weber et al. discloses wherein the mixture is formed into solid objects prior to introducing (col. 6, lines 21-40).

With respect to claims 5, 13 and 20, Weber et al. discloses wherein the coke is discharged as solid objects (molded coke) (col. 6, lines 31-40).

With respect to claims 7, 15 and 22, Weber et al. discloses wherein tar is fed back to the mixture prior to introducing (col. 2,lines 43-46 and col. 7, lines 44-48).

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With respect to claims 8 and 16, Weber et al. discloses wherein separating comprises cooling of pyrolytic by-products by condensing means (fig. 2) into tar (31) and off gas (78).

Claim Rejections - 35 USC § 103

- 5. The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:
 - (a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negatived by the manner in which the invention was made.
- 6. This application currently names joint inventors. In considering patentability of the claims under 35 U.S.C. 103(a), the examiner presumes that the subject matter of the various claims was commonly owned at the time any inventions covered therein were made absent any evidence to the contrary. Applicant is advised of the obligation under 37 CFR 1.56 to point out the inventor and invention dates of each claim that was not commonly owned at the time a later invention was made in order for the examiner to consider the applicability of 35 U.S.C. 103(c) and potential 35 U.S.C. 102(e), (f) or (g) prior art under 35 U.S.C. 103(a).
- 7. Claims 2, 10, 17-20 and 22-27 are rejected under 35 U.S.C. 103(a) as being unpatentable over Weber et al. (4,352,720) as applied to claims 1, 9 and 17 above, and further in view of Deering et al. (4,530,752).

With respect to claims 2 and 10, the process of Weber et al. discloses wherein coal fines and mixtures are introduced (col. 2, lines 10-11) but are silent as to what the mixture comprises.

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Deering et al. teach another similar pyrolyzation process and disclose wherein fines introduced into the retort can be coal fines as well as coke fines (col. 8, lines 50-55). It would have been obvious to one of ordinary skill in the art at the time the invention was made to introduce coke fines as well as coke fines as the starting mixture since it is merely the selection of known starting materials for such as process, additionally, it economically utilizes coke fines that may otherwise be discharged.

With respect to claim 17, Weber et al. discloses a method of producing coke comprising the steps of:

introducing a mixture of coal fines into a pyrolyzer (coking oven) (col. 2, lines 10-19);

pyrolyzing the mixture (col. 2,lines 15-17);

discharging coke (45) and by-products from the pyrolyzer (69);

separating they pyrolytic by-products by condensing means (fig. 2) into tar (31) and off gas (78);

using the tar as a binder in the mixture without discharging to the environment (col. 2,lines 43-46 and col. 7, lines 44-48); and

using the off gas as a source of fuel in the pyrolyzer without discharging to the environment (col. 7, lines 25-30).

The process of Weber et al. discloses wherein coal fines and mixtures are introduced (col. 2, lines 10-11) but are silent as to what the mixture comprises.

Deering et al. teach another similar pyrolyzation process and disclose wherein fines introduced into the retort can be coal fines as well as coke fines (col. 8, lines 50-

55). It would have been obvious to one of ordinary skill in the art at the time the invention was made to introduce coke fines as well as coke fines as the starting mixture since it is merely the selection of known starting materials for such as process, additionally, it economically utilizes coke fines that may otherwise be discharged.

With respect to claim 18, Weber et al. discloses wherein coal is crushed prior to introducing (col. 6, lines 1-4).

With respect to claim 19, Weber et al. discloses wherein the mixture is formed into solid objects prior to introducing (col. 6, lines 21-40).

With respect to claim 20, Weber et al. discloses wherein the coke is discharged as solid objects (molded coke) (col. 6, lines 31-40).

With respect to claim 22, Weber et al. discloses wherein tar is fed back to the mixture prior to introducing (col. 2,lines 43-46 and col. 7, lines 44-48).

With respect to claim 25, Deering et al. disclose wherein the coke breeze (fines) comprises 5-10% weight of the feed mixture (col. 8, lines 58-61).

With respect to claims 23 and 24, Deering et al. recognizes that the amount of coke introduced is a result effective variable (col. 8, lines 58-61) and therefore the amount of the remaining portion of the mixture, coal, is also a result effective variable. As such, without showing unexpected results, the claimed percentage can not be considered "critical". Accordingly, one having ordinary skill in the art at the time the invention was made would have routinely optimized the percentage of coke in the mixture to obtain desired rate and efficiency of operation. *In re Boesch*, 617 F.2d. 272, 205 USPQ 215 (CCPA 1980). And since it has been held that where the general

conditions of a claim are disclosed in the prior art, discovering the optimum or workable range involves only routine skill in the art.

With respect to claim 26, Weber et al. discloses wherein the pyrolyzing act comprises heating within the range of 800-1100°C (col. 2, lines 47-49).

With respect to claim 27, Weber et al. disclose the temperatures of only portions of the cooling system (fig. 2), but it is held that any temperature sufficient to produce tar would have been obvious to one of ordinary skill in the art without undo experimentation and therefore the cooling temperature is held an a result effective variable.

As such, without showing unexpected results, the claimed temperature can not be considered "critical". Accordingly, one having ordinary skill in the art at the time the invention was made would have routinely optimized the cooling temperature to produce tar and obtain desired rate and efficiency of operation. *In re Boesch*, 617 F.2d. 272, 205 USPQ 215 (CCPA 1980). And since it has been held that where the general conditions of a claim are disclosed in the prior art, discovering the optimum or workable range involves only routine skill in the art.

8. Claims 6 and 14 are rejected under 35 U.S.C. 103(a) as being unpatentable over Weber et al. (4,352,720) as applied to claims 1 and 9 above, and further in view of Nicaud et al. (6,043,289).

With respect to claims 6 and 14, Weber et al. discloses combining separated tar (col. 2,lines 43-46 and col. 7, lines 44-48), a binder (bitumen) (col. 7, lines 44-48) and fines mixture (col. 2, lines 10-11) in the introducing act, but is silent as to if synthetic binder is used.

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Nicaud et al. teaches wherein the characteristics of the conventionally obtained binder bitumen are close to those of synthetic bitumen (col. 2, lines 38-42). It would have been obvious to one of ordinary skill in the art at the time the invention was made to select either conventionally obtained bitumen or synthetic bitumen as it is merely the selection of functionally equivalent binders known to the art. Additionally, Weber et al. does not preclude one from using a synthetic binder.

9. Claim 21 is rejected under 35 U.S.C. 103(a) as being unpatentable over Weber et al. (4,352,720) in view of Deering et al. (4,530,752) as applied to claim 17 above, and further in view of Nicaud et al. (6,043,289).

With respect to claim 21, Weber et al. discloses combining separated tar (col. 2, lines 43-46 and col. 7, lines 44-48), a binder (bitumen) (col. 7, lines 44-48) and fines mixture (col. 2, lines 10-11) in the introducing act, but is silent as to if synthetic binder is used.

Nicaud et al. teaches wherein the characteristics of the conventionally obtained binder bitumen are close to those of synthetic bitumen (col. 2, lines 38-42). It would have been obvious to one of ordinary skill in the art at the time the invention was made to select either conventionally obtained bitumen or synthetic bitumen as it is merely the selection of functionally equivalent binders known to the art. Additionally, Weber et al. does not preclude one from using a synthetic binder.

10. Claims 30 and 31 are rejected under 35 U.S.C. 103(a) as being unpatentable over Weber et al. (4,352,720).

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With respect to claim 30, it would have been obvious to one of ordinary skill in the art at the time the invention was made to utilize all of the tar as binder and all of the off gas as fuel in order to efficiently use the products of the reaction as well as to avoid releasing any products into the environment.

With respect to claim 31, it would have been obvious to one of ordinary skill in the art at the time the invention was made to use condensed tar as the sole binder source and off-gas as the sole fuel for the pyrolyzer since both are sources the most readily available binder and fuels since both are products of the reaction, as well as being economical and environmentally sound method of product use.

Conclusion

11. Any inquiry concerning this communication or earlier communications from the examiner should be directed to Alexa A. Doroshenk whose telephone number is 703-305-0074. The examiner can normally be reached on Monday - Thursday from 9:00 AM - 7:30 PM.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Glenn Caldarola can be reached on 703-308-6824. The fax phone numbers for the organization where this application or proceeding is assigned are 703-872-9310 for regular communications and 703-872-9311 for After Final communications.

Any inquiry of a general nature or relating to the status of this application or proceeding should be directed to the receptionist whose telephone number is 703-308-0661.

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ربهم AAD

December 23, 2002

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of:

Craig N. Eatough, et al.

Docket: 8333

Serial No.:09/954,603

Art Unit: 1764

Filed:

September 17, 2001

Examiner:

Alexa A.

Doroshenk

For:

CLEAN PRODUCTION OF COKE

<u>DECLARATION OF L. DOUGLAS SMOOT, PH.D.</u>

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

Sir:

- I. L. Douglas Smoot, declare as follows:
- 1. I am a resident of the State of Utah and a citizen of the United States of America.
- 2. I am over 21 years old and competent to testify on matters set forth in this Declaration. I am a minority owner (member) of the assignee of the above-identified patent application.
- 3. I have a Ph.D. degree from the University of Washington in Chemical Engineering.
- 4. I am skilled in the fuels field including the production of coke. My Curriculum Vitae is attached as Exhibit "A."
- 5. I have been provided a copy of the above-identified application as filed, a copy of two Office Actions respectively mailed December 26, 2002

and August 25, 2003, a copy of U.S. Patent 4,352,720, and a copy of two Amendments, one mailed April 17, 2003 and the other to be mailed contemporaneously with this Declaration.

- determine if the above-identified application provides a written description of the invention as presently claimed. More specifically, I was asked if the present application discloses an apparatus and a process which comprises equipment and/or methodology by which the fines being displaced are washed or elutriated. I conclude, as one having skill in this art, that there is no disclosure of washing or elutriating of the fines being displaced using the equipment and methodology disclosed to produce coke. Thus, in my opinion, it is correct to say the limitations of "unwashed" and "non-elutriated" in the claims are properly supported by the present specification because the fines displacement are neither washed nor elutriated.
- 7. The above-identified patent application is directed to a lower cost way of using coal fines, including discarded coal fines, and coke fines, including discarded coke fines, to produce high grade coke.
- 8. The analysis of Weber (U.S. Pat. No. 4,352,720) found at pages 12-15 of the first Amendment is accurate. Weber discloses an expensive rather than a lower cost way of producing coke. Weber requires expensive equipment by which washed and wet coal from bin 2 is elutriated, by a stream of gas emanating at generator 10, up channel 5 and thence along three paths 20, 21, 23 as dried coal to bin 29.

- 9. Weber's requirements of washed and wet coal and elutriation of fines are contrary to and contradict the concept disclosed and the limitations of the presently claimed subject matter of the above-identified patent application. Specifically, the present application does not disclose displacing washed and wet coal, nor is wet coal elutriated to dry the same prior to pyrolization. The technologies of Weber and the present patent application are very different.
- 10. Given that the present disclosure, which clearly avoids both washing to create wet coal fines and drying of wet coal by elutriation in the production of coke, it is appropriate to distinguish the claimed subject matter from Weber by use of the terms "unwashed" and "non-elutriated." The concept of "unwashed" and "non-elutriated" is unquestionably part of the initially disclosed subject matter of the above-identified patent application, is so understood by me, and would be so understood by anyone with skill in the art.
- 11. The drawings of the above-identified application also make it clear that the displacement of fines through the illustrated equipment does not result in washing of displaced fines or in drying of wet fines through elutriation.
- 12. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with

the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

L. Douglas Smob

Date:

L. DOUGLAS SMOOT Curriculum Vitae

I. SUMMARY

Dr. L. Douglas Smoot is Professor Emeritus of Chemical Engineering, Founding Director of the Advanced Combustion Engineering Research Center at Brigham Young University, and Dean Emeritus of the College of Engineering and Technology. He is also a consultant and expert witness in explosion, combustion, energy, environment and propulsion-related areas.

Dr. Smoot completed his graduate work at the University of Washington in 1960. He has been at Brigham Young University since 1967 and was Department Chairman from 1970 to 1977 and Dean from 1977 to 1994. Previous experience included four years at Lockheed, one year at California Institute of Technology, and summers with Hercules, Phillips Petroleum Company and Boeing. He has also consulted with about seventy companies and agencies in energy, combustion, fires and explosions, and chemical propulsion in the United States, Europe and the Orient. He has been a consultant and expert witness on many legal cases involving a variety of explosions and fires over the past two decades.

While serving as Dean from 1977 to 1994, the college external research budget increased by more than ten-fold, the number of faculty was increased by 35, and college majors more than doubled. He developed and implemented a five-year college development plan, a "profile" method for administering and evaluating departments, an effective teacher development program, and a building program to accommodate the rapid enrollment issue, including the new Crabtree Technology Building.

Since 1968, Dr. Smoot has served as principal investigator or project director at BYU on over \$35,000,000 in combustion-related research from industrial and governmental agencies including The Electric Power Research Institute, U.S. Bureau of Mines, NASA, Lockheed, the Navy Department, the U.S. Department of Energy (METC, PETC, GFPO), Utah Power and Light Co., Los Alamos National Laboratory, the National Science Foundation, Tennessee Valley Authority, Empire State Electric Energy Research Co. and several others. Areas of emphasis have included fossil fuel combustion and pollutant emissions, theory of combustion, rocket propulsion combustion, coal gasification, fires and explosions.

In May, 1986, Dr. Smoot was named the founding Director of the newly-organized Advanced Combustion Engineering Research Center, a joint effort with the University of Utah. This Center was sponsored by the National Science Foundation, the State of Utah, and fifty corporations and agencies in the United States, with an annual budget of about \$5,000,000 and with a goal to develop computer-aided design technology for advanced combustion systems. In this position, he directed the largest research program on the BYU campus which involved forty faculty and professionals and a hundred research students on the two campuses for twelve years.

He is a member of AIChE, ASEE, Combustion Institute and the National Fire Protection Association, and has received six state or regional awards. He has published over one hundred peer-reviewed papers and invited reviews, over 100 other technical articles, four books and five chapters in books on combustion, fires and explosions. He served on the Governor's Science and Technology Advisory Council for the State of Utah. He was given the 1985 Distinguished Faculty

Award at BYU and the same year received the University's Presidential Award. He also received the first Governor's Medal for Science and Technology in the State of Utah in 1987. In 1994, he was confirmed by the U.S. Senate as a congressional advisor to the Office of Technology Assessment. The university awarded Dr. Smoot a professorship in combustion in 1994. In 1995, he received the Outstanding Faculty Award in BYU's College of Engineering and Technology. Also, in 1995-1996, he served as Assistant Commissioner of the Church Educational System, spending part of his time developing humanitarian service opportunities in higher education in developing nations. In October of 2002, the U.S. Department of Energy, Fossil Energy Division, presented its most distinguished award, the Homer H. Lowry Gold Medal, to Dr. Smoot for outstanding contributions to the science and technology of fossil energy. And, in his community of Provo, he has donated over 1000 hours per year for the past eight years in service on three different councils, including chairing of a successful effort to rescue and preserve the historic Brigham Young Academy Building as the home of the city's new library.

II. ACADEMIC RANK, POSITIONS

Assistant Professor, 1960-63
Associate Professor, 1967-69
Professor, 1970-2002
Department Chairman, 1970-77
Dean, 1977-1994
Head, Combustion Laboratory, 1977-1990
Director, Advanced Combustion Engineering Research Center, 1986-1997
University Professorship in Combustion, 1994-2002
Professor Emeritus, 2003-

III. DEGREES AND SCHOOLS

B.S., Chemistry, Brigham Young University, 1957
B.E.S., Chemical Engineering, Brigham Young University, 1957
M.S., Chemical Engineering, University of Washington, 1958
Ph.D., Chemical Engineering, University of Washington, 1960
Special Courses, Chemical Engineering, University of California, Los Angeles, 1965
Special Courses, Chemical Engineering, California Institute of Technology, 1966-67

IV. RELATED EXPERIENCE

| A. <u>Teaching</u> | |
|--------------------|--|
| 1953 | Lab Instructor, Chemistry, Brigham Young University |
| 1954-56 | Instructor, Mathematics, Brigham Young University |
| 1958-59 | Teaching Assistant, University of Washington |
| 1960-63 | Assistant Professor, Brigham Young University |
| 1966-67 | Visiting Assistant Professor, California Institute of Technology |
| 1967-2002 | Associate Professor, Professor, Brigham Young University |
| 2003- | Professor Emeritus, Brigham Young University |

B. Industrial

1956 (summer) Student Engineer, Boeing Airplane Company, Plastics Division, Seattle, Washington

1957 (summer) Process Engineer, Phillips Petroleum Company, Atomic Energy Division, Bacchus, Utah

Senior Development Engineer, Hercules Power Company, Chemical Propulsion Div., Bacchus, Utah 1961-62 1963-67

Senior Technical Specialist, Supervisor, Project Engineer, Program Manager, Lockheed Propulsion

Company, Redlands, California

V. RECENT PUBLICATIONS (1993-2002)

In the past decade, Dr. Smoot has edited and co-authored two new books on combustion, co-authored four combustion chapters in prominent encyclopedias, published six invited review articles, and twenty-two peer-reviewed papers based on original research. This list of publications totals over 230 in the past four decades.

VL BIOGRAPHY LISTINGS

Who's Who in Technology Today Who's Who in the West Who's Who in America American Men and Women in Science

Who's Who in Consulting Who's Who in Aviation Who's Who in Engineering

VII. COMPANIES CONSULTED FOR

Hercules, Inc., Salt Lake City, Utah Thiokol Chemical Corporation, Brigham City, Utah Lockheed, Redlands, California and Sunnyvale, California Teledyne Corporation, Hollister, California Raytheon Corporation, Bedford, Massachusetts Redd and Redd, Salt Lake City, Utah Billings Energy Corporation, Provo, Utah Ford, Bacon and Davis, Salt Lake City, Utah Jaycor, Inc., Del Mar, California Atlantic Research Corporation, Arlington, Virginia Intel Com Radiation Technology, San Diego, California Physical Dynamics, Berkeley, California National Society of Propellants and Explosives, Paris, France DFVLR, West Germany Martin Marietta Corporation, Denver, Colorado Honeywell Corporation, Minneapolis Minnesota National Bureau of Standards, Washington, D.C. Systems, Science and Software, San Diego, California Lockheed Research Laboratories, Palo Alto, California Exxon Corp., Floram Park, New Jersey Los Alamos National Laboratory, New Mexico Phillips Petroleum Co., Bartlesville, Oklahoma Eyring Research Institute, Provo, Utah Utah Power & Light Co., Salt Lake City, Utah U.S. Department of Energy, Washington D.C. Tennessee Valley Authority, Chattanooga, Tennessee U.S. Department of Energy, Morgantown Energy Technology Center Mountain States Energy Co., Bozeman, Montana Energy and Environmental Research Corp., Irvine, California

Science Applications International Corporation, San Diego, California

Argonne National laboratory, Chicago, Illinois

Kirkland And Ellis, Chicago, Illinois

Continuum Corp., Huntsville, Alabama

Bituminous Coal Research Institute, Pittsburgh, Pennsylvania

Empire State Electric Energy Research Corp., New York, New York

Supreme Council of Universities, Cairo, Egypt

Intermountain Fuel Corporation, Salt Lake City, Utah

Science Applications International, San Diego, California

Carbon Fuels Inc., Denver, Colorado

Howard, Lewis and Peterson, Provo, Utah

TRW Corp., North Carolina

ADA Corporation, Aurora, Colorado

Geneva Steel, Provo, Utah

Ray, Quinney and Nebeker, Salt Lake City, Utah

Envirochem Corp., Orem, Utah

Pacific Generation Co., Portland, Oregon

Fisher and Hurst, San Francisco, California

Kimball and Parr, Salt Lake City, Utah

Wilcox, Dewsnup and King, Salt Lake City, Utah

Anderson and Schwab, Inc., New York, New York

Covol, Inc., Lehi, Utah

Combustion Resources, LLC, Provo, Utah

Bell, Metzner, Gierhart and Moore, S.C., Madison, Wisconsin

Venturi Technologies, Orem, Utah

Pacific Corp., Portland, Oregon

Purwox, Inc., Las Vegas, Nevada

MCNIC Pipeline and Processing Co., Detroit, Michigan

Earthco Corp., Indianapolis, Indiana

Keleher and McCloud, Albuquerque, New Mexico

Butkus and Reimer, Albuquerque, New Mexico

Simonsen, Hess and Leibowitz, New York, New York

Broening, Oberg, Woods, Wilson and Cass, Phoenix, Arizona

Holme, Roberts and Owen, Salt Lake City, Utah

Johns and Flaherty, LaCross, Wisconsin

Kasieta Legal Group, Madison, Wisconsin

Dana G. Kirk, Houston, Texas

Crowe and Scott, Phoenix, Arizona

Kemp Law Firm, Dallas, Texas

Mitchell, McNutt, et al., Jackson, Mississippi

Cozen and O'Connor, San Diego, California

Gary C. Mitchell, P.C., Ruidoso, New Mexico

Dixon, Truman and Kelleher, Las Vegas, Nevada

Berg, Simpson, Eldredge and Hersh, P.C., Englewood, Colorado

Several others

VIII. SCIENTIFIC AND PROFESSIONAL SOCIETY ACTIVITIES

Utah, Registered Professional Engineer No. 2732

Member, Sigma Xi and Research Society of America

Member, American Institute of Chemical Engineers

Member, American Society for Engineering Education

Member, Phi Lambda Epsilon

Member, International Tripartite Technical Cooperative Program Attenuation Working Group, 1966-72

Chairman, Ad Hoc Committee on Hybrid Combustion, U.S. Interagency Chemical Rocket Propulsion Group, 1965-67

Chairman, Research Society of American (RESA), Lockheed Propulsion Company Branch, 1967

Chairman, Technical Session, American Institute of Chemical Engineers National Mtg., Los Angeles, California, 1968

Session Chairman, Aerospace Corporation Special Conference on Exhaust Volume Technology, July, 1968

Program Chairman, Brigham Young University Engineering Symposium, 1962-63

General Co-Chairman, National Meeting of 6th Joint Army-Navy-NASA-Air Force Plume Technology Conference Monterey, California, 1967

Member, Papers Committee, International Combustion Institute Symposia, 1968-1980

Board of Directors, Great Salt Lake Section, AIChE, 1972-1973

Co-Chairman, Combustion Fundamentals Sessions, National AIChE Meeting, New Orleans, LA., November, 1981

Governor's Science and Technology Advisory Council, State of Utah, 1979-1982

U.S. Department of Energy, Coal Combustion Applications Working Group, 1982-83

Co-Chairman, Engineering Dean's Institute, Salt Lake City, Utah, March 1982

Member, Editorial Board, Progress in Energy and Combustion Science, 1982-present

Member, Program Committee, International Combustion Symposium, 1984-present

Member, Board of Directors, International Combustion Institute, 1984-1996

Member, Board of Directors, Engineering Dean's Council, 1984-1990

Member, FASAC Committee, for review of Soviet Low Energy Combustion Research, 1985-1986

Member, Advisory Council, Office of Technology Assessment, Washington, DC, 1994-1995.

Member, National Fire Protection Association, 1998

IX. HONORS AND AWARDS

Outstanding Graduate, BYU Chemical Engineering, 1957

Graduated with Highest Honors, Brigham Young University, 1957

Undergraduate Student Award, Outstanding Chemical Engineering Professor, 1970

Panel Member, National Academy of Sciences, Plume Emissions Panel, 1972-73

Outstanding Engineering Educator Award, State of Utah (EJC), 1975

BYU Maeser Research Award, 1975

Outstanding Aerospace Educator Award, State of Utah, 1975

Outstanding Engineer, State of Utah (AIAA), 1977

Educator of the Year, Society of Manufacturing Engineers-Region VII, Western United States, Pasadena, California, October 7, 1978

Outstanding Alumnus Award, Springville High School, Utah, 1980

Distinguished Faculty Lecturer, Brigham Young University, 1985

Presidential Medal, Brigham Young University, 1985

Governor's Medal for Science and Technology, State of Utah, 1987

Chaired Professorship in Combustion, Brigham Young University, 1994

Outstanding Faculty Member, College of Engineering and Technology, 1995

Provo City Reed Smoot Community Service Award, Provo-Orem Chamber of Commerce,

Provo City Citizen of the Year Award, Provo Kiwanis Club, 1997

Utah Heritage Foundation, Heritage Award, 1999

Provo City Citizen of the Year Award, Provo-Orem Chamber of Commerce 2001

U.S. Department of Energy, Homer H. Lowry Gold Medal, 2002

X. RECENT EXPERT WITNESS WORK

1. Howard, Lewis, and Peterson, Provo, Utah

Consultants - Drs. L. Douglas Smoot and Stephen K. Kramer

Firm contact - Mr. Jackson Howard

Case (1984) - Witnessed for the plaintiff who was injured in a dust explosion/fire while cleaning process equipment at a Gilsonite plant. The ignition and explosion characteristics of Gilsonite were identified and compared with coal and other dusts.

2. Howard, Lewis, and Peterson, Provo, Utah

Consultants - Drs. L. Douglas Smoot and Stephen K. Kramer

Firm contact - Mr. Jackson Howard

Case (1989) - Witnessed for the plaintiff where plaintiff was injured while firing a black powder rifle with smokeless powder. The characteristics and burning rate of black powder and smokeless powder were compared. A computer model was developed to simulate effects. Testimony was provided at the trial.

3. Clyde, Pratt, Gibbs, and Cahoon, Los Angeles, California

Consultant - Dr. L. Douglas Smoot

Firm contact - Mr. Lee Pratt

Case (1985) - Witnessed for the defendant in a case concerning a coal dust explosion and fire near a large power generating plant operated by the Nevada Power Company. Settled before trial.

4. Kimball and Parr, Salt Lake City, Utah

Consultant - Dr. L. Douglas Smoot

Firm contact - Mr. Steven Christiansen

Case (1988) - Provided consulting services regarding pollutant emission regulations relating to the Geneva Steel plant in Utah County. Settled before trial.

5. Kirkland and Ellis, Chicago, Illinois

Consultants - Drs. L. Douglas Smoot and Craig N. Eatough

Firm contact - Mr. Robert Krupka

Case (1988) - Witnessed for the defendant in Dow-Texaco trade secret case involving a large, industrial-scale coal gasification process. Different coal-water slurry delivery systems were analyzed and comparisons of similarities and differences in the two gasification processes were considered in light of public domain information. Settled before trial and depositions.

6. Fisher and Hurst, San Francisco, California

Consultants - Drs. L. Douglas Smoot and Stephen K. Kramer

Firm contact - Mr. Roland Stevens

Case (1989-1990) - Witness was provided for the plaintiff concerning the origin and effects of a fire in the Wilberg coal mine that caused the deaths of 27 people. The autoignition and combustion of compressor oil in the air compressor was considered and calculated effects were matched with examination of the failed equipment. A computer model was developed of the crosscut fire and used to provide approximate times that were then matched to observations made by mine employees. Extensive testimony was provided during depositions and the trial.

7. Ray, Quinney, and Nebeker, Salt Lake City, Utah

Consultants - Drs. L. Douglas Smoot and Craig N. Eatough

Firm contact - Mr. Steven Aeschbacher

Case (1992) - Witnessed for plaintiff who was burned while filling a five gallon gasoline can in the back of his pick-up. The possible sources of ignition were evaluated and the likely cause established. Settled before trial and depositions.

8. Wilcox, Dewsnup, and King, Salt Lake City, Utah

Consultants - Drs. L. Douglas Smoot and Paul O. Berrett (EE)

Firm contact - Mr. Brent Wilcox

Case (1994-1995) - Witness for plaintiff who was badly burned in accidental fire during heating of oil prior to truck transport. Provided technical analysis on cause and origin.

9. Anderson and Schwab, New York

Consultants - Dr. L. Douglas Smoot

Contact - Mr. Van Tingley

Case - Expert consultant for U.S. Justice Department accused of preventing development of a gasification project on Wyoming coal mine lands. Provided technical services. Case settled prior to depositions.

10. Wilcox, Dewsnup, and King, Salt Lake City, Utah

Consultants - Drs. L. Douglas Smoot, Stephen K. Kramer, Craig N. Eatough and Scott C. Hill

Firm contact - Mr. Jeffery Eisenberg

Case (1994-1997) - Witness for the plaintiff who was badly burned while laying carpet in the basement of a home while using a volatile contact cement. Fire cause was confirmed and cement conformance with regulations investigated. Provided depositions. Case settled following depositions (September, 1997).

11. Kimball, Parr, Waddoups, Brown and Gee, Salt Lake City, Utah

(Subsequently Johnson and Hatch, Salt Lake City, Utah)

Consultants - Drs. L. Douglas Smoot, Craig N. Eatough and Scott C. Hill

Firm Contacts - Mr. Clark Waddoups, Mr. Paul Drecksel

Case (1997-1998) - Geneva Steel vs. Commerce and Industry Insurance Co. Witness for plaintiff (Geneva Steel Co.) whose coke batteries were allegedly damaged during a power outage. Conducted computerized analysis of pressure and temperature transients. Depositions provided. Case settled, August, 1998.

12. Bell, Metzner, Gierhart and Moore, Madison, Wisconsin

(Subsequently Robert Kasieta Legal Group, Madison, Wisconsin)

Consultants - Drs. L. Douglas Smoot, Craig N. Eatough and Scott C. Hill:

Firm Contact - Mr. Houston Parrish (Subsequently Mr. Robert Kasieta)

Case (1996-1997) - Olsen vs. Northern Engraving, et al. Witness for defendant (AAR Power Boss, Inc.) in an acetone vapor-liquid fire during floor cleaning at an industrial site with a floor scrubber. Provided analyses and opinions on behavior of acetone. Depositions provided. Case settled with defendant following depositions (October, 1997).

13. Butkus and Reimer, P.C., Attorneys at Law, Albuquerque, New Mexico

(Subsequently Kelcher and Mcleod, P.A., Albuquerque, New Mexico)

Consultants - Drs. L. Douglas Smoot, Craig N. Eatough, Brent Strong and David Jensen

Firm Contact - Mr. R. Galen Reimer (Subsequently Mr. M. Charles Pharris)

Case (1997-1999) Witness for the defendant in Langley vs. Maytag Corp. Providing consulting services, fire analysis and testimony for the defendant (Maytag Corp.) on a fire at a Carrizozo, NM ranch house in 1994. Fire allegedly started by overheated freezer. Depositions in January and May, 1998. Case settled, Feb. 1999.

14. Johns and Flaherty, S.C., Attorneys, La Crosse, Wisconsin

Consultants - Drs. L. Douglas Smoot and Scott C. Hill

Firm Contact - Mr. Michael L. Stoker

Case (1997-1998) - Olsen vs. Northern Engraving, et al. Consultant for plaintiff (Mr. Gilbert Olsen) to perform computerized fire-site simulations for the acetone vapor-liquid fire in 1994 during floor cleaning with a scrubber at an industrial site. Case settled before depositions on this part of the case.

15. Holme, Roberts and Owen, LLP, Sait Lake City, Utah

Consultants - Drs. L. Douglas Smoot, Craig N. Eatough and David Jensen

Firm Contacts - Mr. George M. Haley, Mr. Gregory J. Savage

Case (1997-) - Clayton vs. Alpine Supply Co. Witness for plaintiff injured in an explosion of a gas-fired fireplace insert in July, 1997. Conducted analyses of fireplace and incident and reviewed standards. Case active; depositions, August, 1999. Case settled, October 1999.

16. Simonsen, Hess and Leibowitz, P.D., New York, New York

Consultants - Drs. L. Douglas Smoot, Craig N. Eatough, Geoffrey J. Germane and Mr. Robert Jackson

Firm Contact - Mr. Alan B. Leibowitz

Case (1998-) - Hickey vs. Chrysler Corp./Lazar's Auto Sales. Witness for plaintiff on fire/explosion origin and cause during 1990 Jeep Wrangler rollover and fire in April, 1992, near Mt. Kisco, NY, where driver was fatally injured. Case settled, October 1999.

17. Broening, Oberg, Woods, Wilson and Cass, P.C., Phoenix, Arizona

Consultants - Drs. L. Douglas Smoot and Craig N. Eatough

Firm Contact - Mr. James R. Broening, Mr. Bruce Preston

Case (1998-) - ASARCO vs. Geneva Steel, et al. Witness for defendant, Geneva Steel, regarding an explosion in January, 1996, in a slag-cleaning furnace at a copper smelter in Hayden, AZ, allegedly from use of coke breeze product provided by the defendant. Two workers were injured. File completed, deposition, mid 2002; case pending.

18. Dana G. Kirk Law Offices, Houston Texas.

Consultants - Dr. L. Douglas Smoot

Firm Contact - Mr. Dana G. Kirk

Case (1999 -2000) - Witness for Plaintiffs vs. Formosa Chemical Company. Explosion of Ethylene Dichloride plant, injuries reportedly of 45 people. Preliminary information provided, no further action.

19. Crowe and Scott, P.A., Phoenix, Arizona.

Consultants - Drs. L. Douglas Smoot and Dale R. Tree; Mr. Robert E. Jackson

Firm Contact - Mr. Thomas Crowe

Case (1999 -) - Witness for defendants in Federal Government vs. Knight, et al. Blending of diesel fuel with heavier fuel oil for transportation fuel. Criminal case settled:

20. Kemp Law Firm, Dallas, Texas.

Consultants - Drs. L. Douglas Smoot and Scott S. Hill; Mr. Robert Jackson.

Firm Contact - Mr. David O. Kemp

Case (1998-1999) - Witness for plaintiffs in Delta Counseling Associates, Inc. vs. Doss Heating and Air Conditioning Co., Inc., et. al. (No. Civ. 95-21-2). Nighttime fire (Jan. 15, 1993) in counseling center, allegedly caused by improperly installed natural gas furnace; destroyed building in Lake Village, Arkansas. Depositions, August, 1999; case settled, October, 1999.

21. Mitchell, McNutt, Threadgill, Smith and Sams, P.A., Jackson, Mississippi.

Consultants - Drs. Geoffery Germane (Germane Engineering) Scott C. Hill, Craig N. Eatough, L. Douglas Smoot.

Firm Contacts - Miles Parker and Tim Warren

Case (1999 -) Witnesses for plaintiffs. Coal-fired utility furnace exploded in Kansas City, Kansas, Feb. 17, 1999 during furnace planned downtime. Preliminary cause investigation. No complaint filed. Case terminated.

22. Cozen and O'Connor Law Unices, San Diego, California

Consultant - Dr. L. Douglas Smoot

Firm Contact - Mr. Kevin D. Bush

Case (1999 -2000) - Consultant for plaintiff. Explosion of crude oil storage tank in Woods Cross, Utah, on Nov. 12, 1998. Conducted preliminary analysis. No complaint filed. Case terminated after preliminary investigation.

23. Dewsnup, King and Olsen, Law Offices, Salt Lake City, Utah

Consultant - Dr. L. Douglas Smoot

Firm Contact - Mr. Jeffrey D. Eisenberg

Case (1999-2000) - Consultant for the plaintiff. Ammonium perchlorate (AP) in a bag - house cleaning system at an AP manufacturing plant near Cedar City, Utah caught fire during manual cleaning on July 30, 1997, with one worker killed, another seriously injured. Preliminary investigation completed. Case inactive.

24. Gary C. Mitchell, P.C., Law Offices; Ruidoso, New Mexico

Consultant - Dr. L. Douglas Smoot

Firm Contact - Ms. Shawn M. Boyne, Mr. Gary C. Mitchell

Case (2000 -) - Consultant for the plaintiff. Ignition and severe fire inside a moving pickup truck cab on Jan. 24, 1998 where a container of smokeless gun powder was being transported from gunshop to home. Two serious injuries. Complaint filed; some depositions taken. Investigation completed. No further action.

25. Dixon and Truman, Law Offices, Las Vegas, Nevada

Consultant - L. Douglas Smoot

Firm Contacts - Thom Gover, Bryce Dixon

Case (2000 -) - Consultant for the plaintiff. On Feb. 27, 1999, a fire occurred when the defendant was filling small gasoline cans in a lined truck bed; he was seriously injured. Technical work ongoing Site and vehicle inspection were completed. Deposition taken, 2002. Case settled, 2002.

26. Berg, Simpson, Eldredge, Hersh P.C., Englewood, Colorado

Consultant - Dr. L. Douglas Smoot

Firm Contact - Mr. Joseph Kaupie

Case (2000 -) - Consultant for the plaintiff. A direct-vent fireplace exploded in Breckenridge, Colorado at

7000 ft. altitude. Preliminary work conducted. Case inactive.

27. Bues, Gilbert, Phoenix, Arizona

Consultant - Dr. L. Douglas Smoot

Firm Contact - Mr. Joseph B. Pierce

Case (2000 -) - Witness for the plaintiff. Mr. Knight v. U.S. Government. Mr. Knight was acquitted as a defendant in a criminal case wherein he allegedly had fraudulently blended and marketed truck diesel fuel in Arizona that did not meet specifications. Following settlement of criminal case (2000), the plaintiff filed a complaint against the federal government for damages resulting from the federal suit. Technical work on-going. Deposition anticipated. Case pending.

28. Williams, Dailey, and O'Leary, P.C., Portland, OR.

Consultant - Dr. L. Douglas Smoot

Firm Contact - Kathleen M. Dailey

Case (2001) - Consultant for the plaintiff. Plaintiff was injured in head on automobile collision and subsequent fire. Consultant provided preliminary consulting services. No complaint filed.

29. Phelps Dunbar, LLP New Orleans, LA

Consultant - Dr. L. Douglas Smoot

Firm Contacts - Barbara L. Arras, Neil Abramson, Steven LeVine

Case (2001-) - Consultant for the defendant. Union Pacific train derailment near Unice, LA with about 30 cars containing various flammable chemicals derailed with a massive fire occurring. Initial analysis completed, file prepared, deposition given at certification hearing, August, 2001; additional report prepared, fall, 2002; case pending.

30. Strong and Hanni, Law Offices, Salt Lake City, Utah.

Consultants - Dr. Craig N. Eatough, Dr. L. Douglas Smoot

Firm Contact - Michael Ford

Case (2001-) - Consultants for the plaintiff. Homeradiant heating boiler was alleged to have started a fire within the home. Provided case directions to principal consultant. Investigation initiated. Case pending.

- 31. MacArthur Moten, P.C., St. Louis, MO Consultants - Dr. Craig N. Eatough, Dr. L. Douglas Smoot Firm Contact - MacArthur Moten Case (2001-) - Consultants for the plaintiff. Home heating natural gas furnace allegedly leaking carbon monoxide. Plaintiff complained of discomfort. Discussed directions with principal consultant; case pending.
- 32. Edwards, Frickle, Halversen and Ames-Hughes, Billings, MT Consultants Dr. N. Stanley Harding, Dr. L. Douglas Smoot Firm Contact Mr. Roger Frickle Case (2001) Consultants for the plaintiff. Plaintiff was badly burned when a large ash clinker fell and water from the bottom hopper of a utility boiler. Investigation completed. Case settled before trial, 2002.
- 33. Barne, Guzy and Steffon, completed LTD, Minneapolis, MN.

 Consultants Dr. N. Stanley Harding, Dr. L. Douglas Smoot

 Firm Contact Mr. John Buchman

 Case (2002) Consultants for the plaintiff. The plaintiff was badly burned when hot ash fell from inclined walls of wood-burning furnace while furnace was shut down for cleaning. Investigation just initiated. Case active.
- 34. Ellison Nielson Knibbs Zone and Antas, P.C. San Francisco, CA. Consultants Mr. Robert Jackson; Dr. L. Douglas Smoot Firm Contact Mr. Richard M. Ozowski Case (2002-) Consultant for the defendant; accidental fire occurred in a church meeting house during a time when heavy equipment construction was on-going outside. Investigation initiated; preliminary report prepared; case pending.
- 35. Dixon and Truman Law Offices, Las Vegas, NV.
 Consultant Dr. L. Douglas Smoot
 Firm Contact A. Bryce Dixon, general partner.
 Case (2002-) Consultant for the plaintiff. On June 8, 2002, plaintiff was badly burned and two vehicles were destroyed in a fire at the Town Pump station of Townsend, MT. Plaintiff was filling two metal cans in back of pickup truck with plaster bed liner at the time of the fire. Investigation initiated, case active.

United States Patent [19]

Weber et al.

[11] 4,352,720

[45] Oct. 5, 1982

| [54] | PROCESS AND APPARATUS FOR THE |
|------|-------------------------------|
| | PRODUCTION OF MOLDED |
| 1.2 | METALLURGICAL COKE FROM COAL |
| | BRIQUETTES |

[75] Inventors: Heinrich Weber, Recklinghausen; Kurt Lorenz, Hattingen; Horst

Dungs, Herne; Klaus Urbye, Flaesheim, all of Fed. Rep. of

Germany

[73] Assignee: Firma Carl Still GmbH & Co., KG,

Fed. Rep. of Germany

[21] Appl. No.: 201,262

[22] Filed: Oct. 27, 1980

Related U.S. Application Data

[62] Division of Ser. No. 135,112, Mar. 28, 1980, Pat. No. 4,305,788.

[30] Foreign Application Priority Data

Apr. 5, 1979 [DE] Fed. Rep. of Germany 2913666

| [51] Int. CI., | | | CIUB 1/04 |
|----------------|-------|-----------------|--------------|
| [52] U.S. Cl | | 202/1 | 24. 202/228. |
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| 4,050,990 9/1 | 977 Lorenz | 201/6 X |

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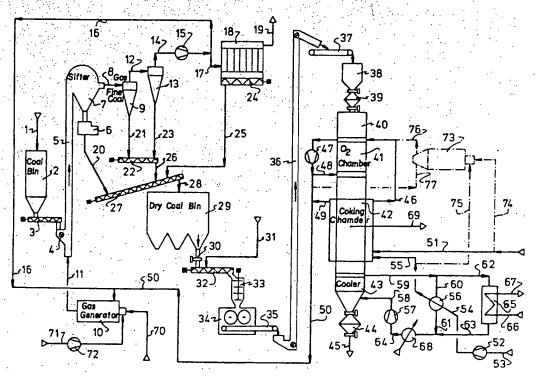
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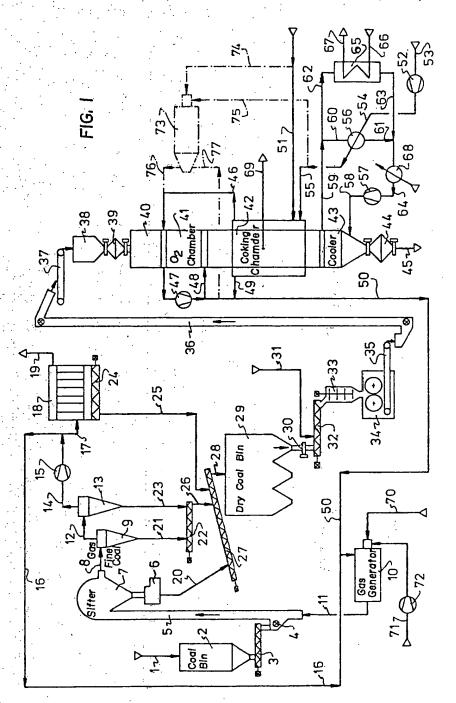
Primary Examiner—Norman Yudkoff
Attorney, Agent, or Firm—McGlew and Tuttle

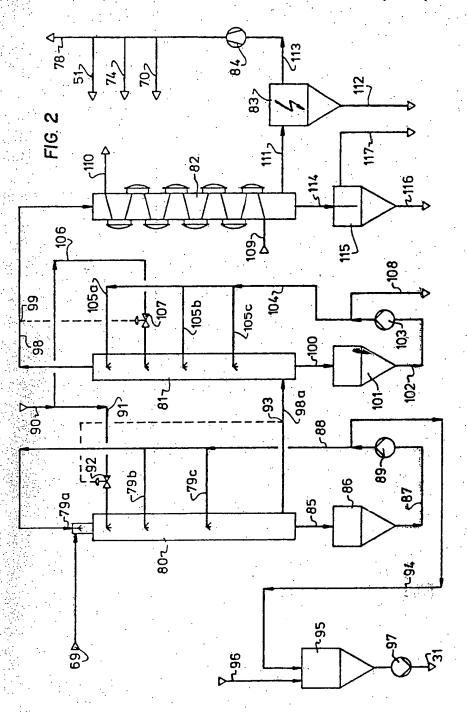
[57] ABSTRACI

The process for the production of molded metallurgical coke from dried coal or fine coal mixtures with a low swelling index particularly highly volatile fine coal comprises mixing normal washed fine coal or fine coal mixtures of a grain size of from 0 to 10 mm and a swelling index of not more than 5 with a binder, pressing the mixture into briquettes, oxidizing the briquettes in a continuous material flow stream, and coking the oxidized briquettes continuously in an oven chamber by supplying indirectly and exhausting the coke oven gases with a temperature of 300° to 1200° C. The device for carrying out the process includes a vertically extending reactor housing having an oxidation chamber adjacent the top thereof with an indirectly heated vertical coking chamber directly below the oxidation chamber and with a cooling chamber directly below the coking chamber and including means for circulating heating gases into the coking chamber and then upwardly through the coking chamber and out adjacent the top of the coking chamber and into the oxidation chamber.

3 Claims, 2 Drawing Figures







PROCESS AND APPARATUS FOR THE PRODUCTION OF MOLDED METALLURGICAL COKE FROM COAL BRIQUETTES

This is a division of application Ser. No. 135,112 filed Mar. 28, 1980, now U.S. Pat. No. 4,305,788, issued Dec. 15, 1981.

FIELD AND BACKGROUND OF THE INVENTION

The invention relates in general to metallurgical operations and in particular to a new and useful device and process for the production of molded metallurgical coke from coal briquettes which are mixed from dried fine coals or fine coal mixtures with a low swelling index, particularly highly volatile fine coals, with a binder, and pressed to briquettes, rehardened, oxidized, coked and cooled.

A process for the production of molded metallurgical coke from highly volatile fine coal and a low swelling index is known, where the coal must be ground very finely, at any rate below 1 mm. After rehardening and oxidation, if any, of these briquettes which are produced from such coals with a binder, the coking is effected by direct heating, that is, by conducting hot combustion gases over the briquettes. A gas with a low calorific value is obtained which has only limited applications. A large amount of low-grade lean gas is obtained this way from the highly volatile coals. This also reduces the economy of the process. Direct heating causes a great loss of carbon by gasification or combustion, namely about a 4% loss.

In another known process for the production of molded metallurgical coke from highly volatile coal with a low swelling index, the coking of the "green" briquettes which can be subjected, if necessary, to an oxidizing after treatment is effected discontinuously 40 with indirect heat transfer.

In this process, as in the conventional chamber coking process, no measures to prevent emissions are provided or possible. Particularly during the filling of the coal, as well as during the discharge, cooling or quench-45 ing of the coke, emission of dust-containing gases, which contain partly combustible or harmful components, are unavoidable.

Emissions also appear to a greater extent because coke cakes frequently at the bottom end, that is, at the outlet of the oven chambers. The cakes must then be crushed by hand with great effort. Dust is produced, which is carried into the atmosphere. Besides, it is very difficult and expensive during the operation of the plant to seal filling hoppers, uptakes and chamber oven doors to such an extent that no gases with tar components can escape:

The above-mentioned methods are described in detail in the report BF V 31 096 of the Battelle Institute 60 Frankfurt e.V.

Presently known methods have thus either a high heat consumption with long coking times and considerable emissions, or there are great carbon losses and only a low-grade lean gas is obtained which has an unfavorable effect on the economy of the operation, particularly when coal with high portions of volatile components is used.

SUMMARY OF THE INVENTION

The invention provides a new process for the production of molded metallurgical coke from dried coal or coal mixtures with a low swelling index, which can be carried out continuously and where no emissions appear and the heat consumption for the coking is much lower compared to the known methods, and where the specific output is higher than in known methods.

10 For the solution of this problem it is suggested to mix normal, washed, fine coal or fine coal mixtures; with a grain size of 0 to 10 mm and a swelling index of not more than 5 in the dry state, with binders and use them for the production of briquettes, and to oxidize the briquettes in the continuous material current, and to coke them then continuously in an oven chamber under indirect heat supply, and to exhaust the coke oven gas from the oven chamber with a temperature of 300° to 1200° C.

This continuous oxidation and coking can be so effected with simple means that emissions of gases to the outside are avoided. It suffices to this end to control the operation so that there is only a slight excess pressure at the point where the briquettes enter the process, and also at the outlet. In the coking zone of the coking chambers the excess pressure is about 5 to 15 m bar.

The process according to the invention is not limited, like the known methods, to medium and highly volatile fine coal with a low swelling index; according to the invention, coal mixtures of all types can be coked. Coals which have by themselves a higher swelling index can be so adjusted by the addition of other types of coal with a low swelling index, or by the addition of coke fines or petroleum coke, that the mixtures have a swelling index of not more than 5.

It is thus practically possible to coke any type of coal. According to one embodiment of the invention, the coke oven gases are exhausted from the oven chamber with a temperature of 300° to 450° C. In this case the heat consumption for the coking is lower, compared to conventional coking methods, and the yield of higher grade excess coke oven gas is higher. Tars are also obtained which are used, as described more fully below, partly as binders for the coal briquettes in the coking process itself.

According to another embodiment of the invention, the coke oven gases are exhausted with a temperature of 800° to 1100° C. No tars and hydrocarbons and no phenols are obtained, because they are cracked before they leave the coking chamber. But no water of formation is obtained either because it is used up in the water reaction taking place in the oven chamber. If the gases are cooled indirectly as a result, there is no waste water problem. Naturally a gas with a somewhat lower calonific value is obtained, since the hydrocarbons, like methane, are cracked, and more hydrogen and carbon monoxide are contained instead in the coke oven gas.

The waste heat of the indirect heating phase can now be used in a known manner, e.g. for preheating the fine coal or in other stages of the process, e.g. for the production of superheated steam, for the storage bin for the briquette-binder, for example, if pitch-type binders are used.

The waste gases from the heating phase, which have a temperature of about 600° C, still contain about 2 to 6% oxygen. For this reason they are used according to the invention for the oxidation of the coal briquettes to

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be coked. This way the process according to the invention becomes particularly economical.

If the oxygen content of the waste gases from the heating phase is less than 2% by vol, in some cases, air is added preferably by means of air preheated by the 5 heat from the process, so that they can be used as oxidation gases.

In the process according to the invention, a large amount of gas of high calorific value is generally obtained, a part of which can be used in the process itself 10 for heating purposes. Beyond that excess gas of high calorific value is available for free disposal.

In order to prevent harmful emissions, the gas used in the process itself for heating purposes and for the production of oxidation and coal drying gases, is worked 15 up and purified.

The process according to the invention can be so carried out that harmless waste gases from the heating phase can issue only at a single point, namely at the gas outlet of the coal predrying station. The cooling gases 20 for the hot coke can be circulated over heat exchangers. They then give off their heat to the combustion air, for example, which is required for heating the coking chamber and also for heating the oxidation gas and there is still sufficient waste heat available to generate 25 the steam required for the subsequent purification of the coke oven gas.

But it is also possible according to the invention to use a specially prepared or existing lean gas for the indirect heating in the coking process, e.g. blast furnace 30 gas so that the total amount of the valuable coke oven gas from the coking process is available for other purposes.

In carrying out the process, the pressures in the oxidation stage are so adapted to the coking stage that only 35 a small amount of valuable coke oven gas passes from the coking stage into the oxidation stage or vice versa; from the oxidation stage to the coking stage. Substantial gas losses are thus avoided.

As a link between the two stages can be provided, 40 e.g. an impedance

According to another embodiment of the invention, a high-boiling tar fraction is used as a binder for the production of the briquettes which is mostly obtained in the first cooling stage to bring about the cooling of the coke 45 oven gas with tar.

The coke oven gases leaving the oven chamber with about 300° to 450° C. are preferably cooled in three stages, in the first stage; mainly with circulating tar and only a small amount of water which evaporates completely and thus leaves no aqueous condensate. The tar obtained in this first cooling stage, which boils above 280° C., also contains almost the entire coal dust which is discharged from the coking chamber with the gas. The excess from this cycle of high-boiling dust-containing tar is preferably used without further preparation as a briquette binder in the process itself.

In the second cooling stage, the gas is cooled exclusively with circulating tar boiling under 280° C. to a temperature of 10° to 50° C. above the water dew point. 60 Aqueous condensate is thus not yet obtained, so that large portions of the tar obtained are anhydrous and dust-free. Then it is further cooled, prepared and purified according to known methods.

Due to the preliminary cooling with tar in the process 65 according to the invention, only a relatively small amount of aqueous condensate is obtained. This is particularly the case when the final cooling of the gas in the

third stage is effected indirectly with cooling water. The device for the further cooling of the gas, particularly the tank separating the tarry and aqueous conden-

sate, can therefore, be made correspondingly small.

If briquettes are used from a coal which requires no oxidation the oxidation stage can be carried out as a simple preheating stage.

The second and third cooling stage can also be combined to one stage, where the cooling of the gases under 250° C. can be effected according to known methods or in known devices, e.g. in cross tube coolers with preceding water atomization.

For controlling the temperature in the coking stage and also for setting a temperature gradient, a part of the waste gas from the heating phase can be returned, with advantage, in a cycle into the heating phase of the coking stage.

Because of the great excess of gas, it is not absolutely necessary to use the waste gases from the heating phase as oxidation gases. Without markedly reducing the economy of the process, a part of the coke oven gas or another fuel can be burnt, e.g. in a special combustion chamber, and the oxygen desired for the oxidation can be adjusted in the waste gas from the combustion, it is particularly advisable to circulate the oxidation gases.

The oxygen content can be easily adjusted. The combustion chamber is preferably designed as a separate and independent part. This procedure and arrangement is particularly advisable when different coals or coal mixtures of varying composition are used, and the operation of the plant must be varied with regard to the temperature, amount, and oxygen content of the oxidation gas, and be adapted to the coal or coal mixture.

For carrying out the process it was found advisable to arrange the oxidation chamber above the indirectly heated vertical oven chamber provided with heating flues. This way the two parts of the plant can be combined to a unit. The flues for the coke oven gas can be arranged, e.g. on the end faces of the oven chambers or along the longitudinal walls of the oven chamber as channels which are open at the bottom. They are preferably arranged at a level which corresponds to three quarters of the chamber height, measured from the base of the heating flue.

This embodiment is preferred when the coke oven gases are to be exhausted with a temperature of up to 450° C.

If the coke oven gases are to be exhausted with a temperature of 800° to 1100° C., it is advisable to arrange the gas drains of the coking chamber in the lower third of the heated chamber height, measured from the base of the heating flue.

Any type of press can be used for the production of the briquettes, e.g. roll presses, ring roll presses, edge runner presses, or extrusion presses. Roll presses are preferred, however, at this point because of their high throughput capacity.

In order to prevent emission practically completely, the oxidation chamber and the oven chamber are surrounded by a common jacket, e.g. a steel plate jacket and the inlet for the briquettes into the oxidation chamber and the outlet for the coke briquetted from the cooling chamber are secured by double locks.

According to the state of the art, all intermediate movements of the fine coal, of the binder, of the coal briquettes, and of the finished coke briquettes are reduced to the necessary minimum.

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Accordingly it is an object of the invention to provide a process for the production of molded metallurgical coke from dried coal or fine coal mixtures with a low swelling index and particularly from highly volatile fine coal comprising mixing normal washed fine coal or 5 fine coal mixtures of a grain size of from 0 to 10 mm and having a swelling index of not more than 5 with a binder, pressing the mixture into a briquette, oxidizing the briquettes in a continuous material flow stream, and coking the oxidized briquettes continuously in an oven 10 chamber by supplying heat indirectly to the chamber and exhausting coke oven gases with a temperature of from 300° to 1200° C.

A further object of the invention is to provide a device for carrying out the process of producing a molded 15 metallurgical coke which includes a vertically extending reaction chamber having an oxidation chamber adjacent the top thereof and indirectly heated vertical coking chamber below the oxidation chamber and a cooling chamber below the indirectly heated vertical 20 coking chamber with means for circulating heating gases into the coking chamber and through the coking chamber and then outwardly and then into the oxidation chamber.

A further object of the invention is to provide a de-25 vice for coking which is simple in design, rugged in construction and economical to manufacture.

The various features of novelty which characterize the invention are pointed out with particularity in the claims annexed to and forming a part of this disclosure. 30 39 For a better understanding of the invention, its operating advantages and specific objects attained by its uses, reference is made to the accompanying drawings and descriptive matter in which preferred embodiments are illustrated.

BRIEF DESCRIPTION OF THE DRAWINGS

In the Drawings:

FIG. 1 is a schematic representation of a plan for carrying out the process for the production of molded 40 metallurgical coke in accordance with the invention; and

FIG. 2 is a diagrammatical view of a gas cooling and purification plan and a tar cycle to supplement the process of FIG. 1.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Referring to the drawings in particular, the invention embodied therein comprises a process for the production of molded metallurgical coke from dried coal or fine coal mixtures with a low swelling index and particularly highly volatile fine coal. In addition the invention includes a device for carrying out the process.

The quantitative data of the numerical example de- 55 scribed below on the basis of the two diagrams refer to an hourly throughput for a large-scale operation.

An amount of 100 metric ton washed fine coal with the following properties: grain size equal to or less than 10 mm, volatile components b 28% waf, input moisture 60 10%, ash content 6%, are fed to a wet coal bin 2 over feed belt 1 and from there over a conveyor-type dosing weigher 3 and feed centrifuge 4 to the fly current drier. 5 in which the coal is dried by an inert gas current (500° C. ab. 91,000 m³n) fed from a hot gas generator 10 and 65 line 11, to a residual water content of less than 1%. The units "m³n" are cubic meter at standard temperature and pressure.

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Only a part of the coal is ground in a hammer mill 6 following fly current drier 5, namely the part with a grain size of over 3 mm deposited at the head of the drier over sifter 7. The fly current drier is therefore, recommended as a particularly suitable drier. Naturally any other type of coal drier can be used (e.g. fluid bed, turbine, rotary drum drier, etc.).

binder, pressing the mixture into a briquette, oxidizing the briquettes in a continuous material flow stream, and coking the oxidized briquettes continuously in an oven 10 from there over line 12 to a series-connected multiple dust arrester 13, and is returned over line 14, enhanced by fan 15, at a temperature of about 170° C. partly through line 16 (25,000 m³n) to hot gas generator 10 and partly through line 17 into a bag filter 18, from there over line 12 to a series-connected multiple dust arrester 13, and is returned over line 14, enhanced by fan 15, at a temperature of about 170° C. partly through line 17 into a bag filter 18, from there over line 12 to a series-connected multiple dust arrester 13, and is returned over line 14, enhanced by fan 15, at a temperature of about 170° C. partly through line 17 into a bag filter 18, from where about 78,000 m³n purified waste gas are released over 19 into the atmosphere.

The predried coal is filled from hammer mill 6, material separator 9, multiple dust arrestor 13 and bag filter 18 over the outlet 20, 21 and 23 and conveyor screws 22, 24 and 27 and outlets 25, 26 and 28 into dry coal bin 29 in an amount of 90 tons at 120° C. The coal is fed over bin outlet 30 to a mixing screw 32 where it is mixed with about 6 tons binder (pitch, tar bitumen from butuminous coal and/or petroleum bitumen) from supply 31, and the mixture is shaped after treatment in a kneading machine 33 in a double roll press 34 to "green briquettes". The briquettes (96 metric tons, 120° C.) are brought to charging bin 40 by suitable conveyor devices 35 to 37 and filling buckets 38 and over lock chamber 39

In a system closed by the two lock chambers 39 and 44, the briquettes slide continuously first through oxidation chamber 41, in which they are oxidized on the surface by hot flue gases with an oxygen content between about 2 to 6%, and through the indirect heated coking chamber 42 and then through cooling chamber 43 in which the hot molded coke of 1000° is cooled to 50° C. directly by a separate cooling cycle. 70 metric tons molded coke then leave the plant through lock chamber 44 and outlet 45.

For the oxidation of the green briquettes, about 75 to 80% of the combustion gases are removed from the indirect heating system of the coking stage through line 46 with 600° C. and blown in a cycle into the briquette charge enhanced by fan 47, through line 48 (290° C., 110,000 m³n). The remaining 25 to 30% waste gases from the indirect heating system are combined over line 49 with the flue gases conducted through the oxidation chamber and conducted over return 50 with 360° C. in an amount of 54,000 m³n to the hot gas generator 10 of fly current drier 5.

For the indirect heating of coking chamber 42, 9000 m³n dry purified coke oven gas are supplied through line 51. The required combustion air (48,000 m³n) is blown from the bottom through line 55 into the heating system of coking stage 42 by means of air blower 52 and lines 53/54 after heating in heat exchanger 56 (from 20° to 500° C.) which is connected with cooling stage 43.

In the cooling cycle, 120,000 m³n are circulated by blower 57. The cooling gas is conducted through line 58 with 30° C. into cooling stage 43 and leaves the latter through line 59 with 600° C. About 40% thereof are conducted over lines 60/61 through heat exchanger 56 to preheat the combustion air, the balance serves to generate 23 t satuated steam with a pressure of 40 bar at 150° C. in the waste heat boiler 65 with steam tubes 66/67, and is cooled together with the 40% from heat exchanger 56 in an auxiliary cooler 68 from 200° C. to

the inlet temperature of the cooling stage of 30° C., and fed to blower 57 through line 64.

The crude gases formed in coking are removed on the end face of the coking shaft at about 75% of the heated height with 350° C. over line 69 and conducted to the 5 gas cooling and purifying station shown in FIG. 2 with the respective tar cycle. The crude gas outlet is so dimensioned that all tar portions in the crude gas are vaporous to prevent condensation of higher boiling tar portions in the colder briquette charges which could 10 lead to clogging. Of the crude gas formed during coking, an amount of 30,000 m³n remains after purification of which 9000 m³n are required for heating coking stage 42. 2200 m³n gas are supplied through line 70 to hot gas generator 10 together with 10,000 m³n combustion air 15 through line 71 and blower 72. 18,000 m³n gas are thus in excess. They have a lower or net calorific value of $Hu+4300 \text{ kcal/m}^3n$.

FIG. 1 also indicates by broken lines the possibility of producing with a special combustion chamber 73 a 20 means for forming briquettes; special oxidation gas with an adjustable oxygen supply by supplying the purified coke oven gas through line 74 and the respective air through 75 and circulating it over line 76/77 in oxidation stage 41...

The crude gas obtained during coking, is conducted 25 successively over 69 for cooling as shown in FIG. 2 through the three cooling stages 80/81/82 before it is available, after liberation of fine dusts in electrofilter 83 and a pressure increase in gas exhauster 84 for drying, oxidation, coking and an excess (line 78).

The cooling of the crude gases is effected in the first cooling stage 80, a tar scrubber, in direct flow mainly with tar, which is circulated and charged through lines 79a, 79b, 79c from the top and at various levels into the scrubber and runs off at the bottom through line 85 into 35 receiver 86 and is circulated through lines 87/88 with pump 89. At the same time such a small amount of water is added to the cooling system through line 90/91, controlled by valve 92 in dependence on the gas temperature measured at 93 at the transition from the first to the 40 second cooling stage that complete evaporation of the water is ensured. Behind pump 89, about 3.5 metric tons excess crude tar are taken from the cycle and fed through line 94 to a mixing tank 95. Together with 2.5 metric tons bitumen added from outside the system 45 through line 96, the required 6 metric tons binder are then conveyed with pump 97 through line 31 for briquetting.

The crude gas is introduced into the second cooling stage 81 at the bottom at 98a and conducted through 50 line 98 with about 120° to 140° C. at the top to cross tube cooler 82. In a similar manner as in the first stage, the crude gas is cooled in the second stage by circulating tar and additional water (line system 99 to 107) in counter flow. At 108 a higher boiling tar is obtained 55

which is to a great extent free of dust and water. From cross tube cooler 82 of the final cooling stage with cooling cycle 109/110 the gas flows with about 30° C. through line 100 to electrofilter 83, from which the remaining fine dust is removed over 112. The purified rich gas from flows through line 113 and exhauster 84 to the points of consumption. The tar obtained on cross tube cooler 82 and the remaining condensate flow over 114 into separator 115 and are obtained separately as an aqueous phase at 116, and as tar oils at 117.

While specific embodiments of the invention have been shown and described in detail to illustrate the application of the principles of the invention, it will be understood that the invention may be embodied otherwise without departing from such principles.

What is claimed is:

1. A device for the production of molded metallurgical coke from dried coal or fine coal mixtures with a low swelling index, comprising:

a vertically extending reactor with a common jacket having an oxidation chamber adjacent the top thereof for receiving the briquettes, an indirectly heated vertical coking chamber directly below the oxidation chamber and a cooling chamber disposed directly below the coking chamber for directly cooling the briquettes using a cooling gas;

means for circulating heating gases around said coking chamber and then upwardly across said coking chamber for indirect heating thereof, and into said oxida-

tion chamber:

a top double air lock chamber connected to a top of said oxidation chamber for admitting the briquettes; and

a bottom double air lock chamber connected to a bottom of said cooling chamber for discharging briquettes;

said means for circulating heating gases operating to supply the heating gases from around said coking chamber to said oxidation chamber with an oxygen content of about 2% to 6% to surface oxidize the briquettes directly;

said coking chamber having an outlet for crude coke gas.

2. A device according to claim 1 wherein said coking chamber has a gas outlet at a level corresponding to \$ of the height of said coking chamber.

3. A device according to claim 2 including crude gas purification means connected between said coking chamber outlet and an inlet to said means for circulating heating gases, for supplying purified crude coke gas to indirectly heat said coking chamber, said means for circulating heating gases including an additional inlet for receiving combustion air to be combined with said purified crude coke gas

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United States Patent [19]

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| [54] | OIL SHAI | E RETORTING PROCESS |
|------|------------|---|
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[21] Appl. No.: 622,638

[22] Filed: Jun. 20, 1984

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|------|-----------------------|------------------------|
| [51] | Int. Cl. ³ | C10G 1/00; C10B 53/06 |
| [52] | U.S. Cl | 208/11 R: 201/28 |
| [58] | Field of Search | 208/8 R, 11 R; 201/28. |
| | • | 201/31, 32: 48/197 R |

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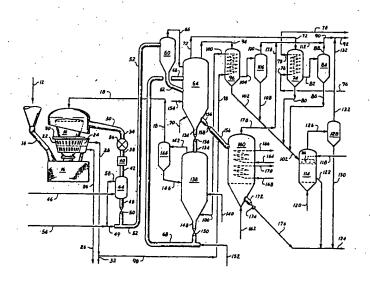
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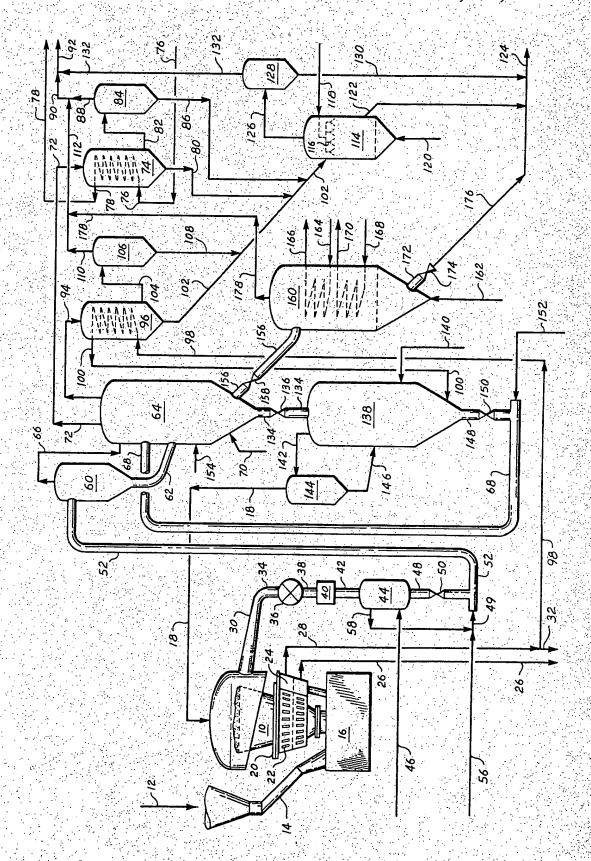
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[57] ABSTRACT

Oil shale solids are pyrolyzed or retorted by contact with a non-oxidizing recycle gas in a first retorting zone to produce an effluent containing pyrolysis products and pyrolyzed oil shale solids. Liquid and gaseous product hydrocarbons are recovered from the effluent of the first retorting zone. The pyrolyzed oil shale solids are contacted with an oxygen-containing gas in a combustion zone under conditions such that at least a portion of the organic material in the pyrolyzed solids is burned to produce hot, decarbonized solids and a hot flue gas. At least a portion of the gases in the effluent from the first retorting zone is contacted in the substantial absence of molecular oxygen with a portion of the hot, decarbonized solids produced in the combustion zone in the presence of added hydrocarbon-containing fines, preferably oil shale fines, in a second retorting zone such that the gases are heated and the fines are pyrolyzed to produce pyrolyzed fines and pyrolysis products containing gases and vapors. The gases and vapors in the effluent from the second retorting zone are then recycled to the first retorting zone where they serve as the hot, non-oxidizing recycle gas.

17 Claims, 1 Drawing Figure





OIL SHALE RETORTING PROCESS

BACKGROUND OF THE INVENTION

This invention relates to the pyrolysis of kerogencontaining oil shale and is particularly concerned with an oil shale retorting process in which the retorted shale is burned to produce hot, decarbonized solids that are used to supply heat for retorting shale fines and for heating recycle retort gas.

Many methods for recovering oil from kerogen-containing oil shale have been proposed in the past. The majority of these methods involve pyrolysis which is commonly referred to as retorting. To be competitive with the production of oils from petroleum stocks, it is 15 essential to recover as much of the heat value from the organic material in the oil shale without incurring prohibitive expense or environmental damage. Normally, oil shale contains between about twenty and about eighty gallons of oil per ton and only a limited propor- 20 tion can be recovered as product oil or gas. Economical retorting must utilize the remaining heat energy contained in the shale to provide heat for pyrolysis. Sulfur emissions in the gases released from the retorting process, however, must be restricted to low levels required 25 by law while attempting to utilize more of the organic material in the shale.

It is known to retort oil shale by a technique of contacting upward flowing, hydrocarbon-containing solids with downflowing gases in a vertical retort. One such 30 technique is disclosed in U.S. Pat. No. 3,361,644, the disclosure of which is hereby incorporated by reference in its entirety. To produce product vapors, the upwardmoving bed of shale particles exchanges heat with a downflowing, hydrocarbonaceous and oxygen-free 35 ble portion of energy available from the shale is wasted. eduction or retorting gas of high specific heat introduced into the top of the retort at a temperature between about 950° F. and about 1200° F. In the upper portion of the retort, the hot eduction gas pyrolyzes the shale, thereby producing hydrogen and hydrocarbonaceous vapors. In the lower portion of the retort, the eduction gas preheats the ascending bed of shale particles to retorting temperatures. As preheating continues, the eduction gas steadily decreases in temperature, condensing high boiling hydrocarbonaceous vapors into a 45 raw shale oil product while leaving a product gas of relatively high BTU content. The shale oil and product gas are then separated, and a portion of the product gas is heated and recycled to the top of the retort as the eduction or retorting gas.

To minimize the volume of recycled gas required, upflow retorting is usually conducted at pressures above atmospheric with the pressure in the upper regions of the retort normally ranging between about 5 p.s.i.a. and about 100 p.s.i.a., preferably between about 55 25 p.s.i.a. and about 65 p.s.i.a. The operation of the retort at superatmospheric pressure, however, means that provisions must be made for introducing and recovering particulate shale from the retorting zone without allowing valuable product and recycle gases to 60 depressure. Conventional methods for achieving these objectives use elaborate lock vessels, valves, or slide valves which tend to wear rapidly and produce excessive fines by abrading the shale. Alternatively, liquid sealing devices, such as described in U.S. Pat. No. 65 4,004,982, have been employed. These devices operate by moving shale particles through a standing head of oil or water, thereby creating a positive back pressure to

prevent escape of retort gases. Liquid seals effectively contain retort gases but leave the shale wet. When incorporated into a process for combusting retorted shale in a vessel separate from the retort, use of liquid seals would require the expense of drying the shale prior to combustion.

To increase product yield beyond that which can be educed in the retort alone, processes have been developed to generate product gases by reaction of hot, retorted shale with an oxidizing gas stream, for example, as taught in U.S. Pat. No. 4,010,092, the disclosure of which is hereby incorporated by reference in its entirety. Such gasification reactions conducted in an oxidizing environment, however, burn the coke on the retorted shale at temperatures high enough to release significant amounts of carbon dioxide from decomposing carbonates in the shale. This, in turn, necessitates expensive removal of carbon dioxide from the combustible product gases. Another source of product yield is unretorted shale fines. Oil shale mined for the purpose of retorting in above-ground retorts is usually crushed mechanically to a size suitable for retorting, normally a top size of about 3 inches or smaller. Due to the friable nature of shale, fines ranging in size up to about & inch are generated in the mining and crushing of larger particles in amounts up to about 10 weight percent of the total shale mined. In above-ground retorting processes, fines mixed with the feed of larger, retort-size particles tend to fill the void spaces between the larger particles. As a result, circulation of hot eduction gases is restricted thus reducing the retort throughput and its oil producing capacity. When the fines are segregated from the feed to the retort to avoid this problem, an apprecia-

Retorted shale contains heat energy in the form of coke which can be recovered by passing the retorted shale particles through a combustion zone to burn the coke Retorted shale, however, generally contains sulfur components and less than complete combustion of the coke generates hydrogen sulfide, which must be removed from the flue gases by means of costly sulfur recovery processes. On the other hand, complete combustion may result in flue gases containing unacceptable quantities of sulfur dioxide. U.S. Pat. No. 4,069,132 discloses a combustion process wherein sulfur dioxide generated during the complete combustion of coke on retorted shale is converted to stable inorganic salts by reaction with alkaline ingredients in the shale. This process utilizes a combustor through which hot retorted shale passes co-currently with air diluted by sufficient flue gas to control peak combustion temperature below about 1670° F. Under such conditions, the discharge of sulfur dioxide from the combustor is disclosed to be greatly reduced.

Because flue gases from combustion zones associated with shale retorts are usually at high temperatures, many retorting processes are designed to utilize the heat contained therein. One example is taught in U.S. Pat. No. 4,069,132, which discloses a process in which hot flue gases are passed in indirect heat exchange with boiler feed water to generate process steam.

Even though many of the above-discussed features have been incorporated into oil shale retorting processes, the need still exists for further developments to improve the efficiency of the processes by effectively retorting raw shale fines using heat generated in the

process while controlling the emissions of hydrogen sulfide and other undesirable gases into the atmosphere.

Accordingly, it is one of the objects of the present invention to provide a process for recovering oil and gas from raw shale fines by retorting the fines utilizing 5 the heat contained in decarbonized solids produced by combusting pyrolyzed shale particles. It is another object of the invention to provide a process to heat the recycle retort or eduction gas used for pyrolysis in the retort utilizing the heat contained in decarbonized solids 10 produced by combusting pyrolyzed shale particles. It is yet another object of the invention to utilize decarbonized solids produced by combusting retorted shale particles to remove a large proportion of hydrogen sulfide and carbon dioxide from the gases produced during retorting in order to minimize processing requirements for product gas clean up. These and other objects of the invention will become more apparent in view of the following description of the invention.

SUMMARY OF THE INVENTION

In accordance with the invention, it has now been found that hot, decarbonized solids produced by combusting retorted oil shale solids can be used to directly provide the heat required to pyrolyze hydrocarboncontaining fines, preferably oil shale fines, while at the same time heating the recycle retort gas used to pyrolyze the oil shale feed solids and removing hydrogen sulfide and carbon dioxide therefrom. Kerogen-containing oil shale solids are contacted with a hot, non-oxidizing recycle gas under pyrolysis conditions in a first retorting zone to produce an effluent containing pyrolysis products and pyrolyzed oil shale solids. Liquid hydrocarbons are recovered from the effluent and the 35 pyrolyzed oil shale solids are passed to a combustion zone. Here the pyrolyzed solids are contacted with a gas containing molecular oxygen under conditions such that at least a portion of the organic material remaining in the solids is burned to produce hot, decarbonized 40 solids and a hot flue gas. A portion of the hot, decarbonized solids produced in the combustion zone is then contacted with gases recovered from the effluent of the first retorting zone in the substantial absence of molecular oxygen in a second retorting zone in the presence of 45 added hydrocarbon-containing fines. The heat from the decarbonized solids is sufficient to pyrolyze the hydrocarbon-containing fines to produce pyrolysis products containing gases and vapors and at the same time heat the gases recovered from the effluent of the first retort- 50 ing zone. The gases and vapors in the effluent from the second retorting zone are then recycled to the first retorting zone for use as the hot, non-oxidizing recycle

In a preferred embodiment of the invention, the gases 55 recovered from the effluent of the first retorting zone are passed in indirect heat exchange with at least a portion of the hot flue gas produced in the combustion zone to preheat the retort gas prior to contacting the gas with a portion of the hot, decarbonized solids produced 60 during combustion. The performance of this additional step results in more efficient use of the heat energy generated in the overall process.

BRIEF DESCRIPTION OF THE DRAWING

The drawing is a schematic flow diagram of a process for retorting oil shale carried out in accordance with the invention.

DETAILED DESCRIPTION OF THE INVENTION

The process depicted in the drawing is one for the retorting of kerogen-containing oil shale solids to produce pyrolyzed solids which are subsequently combusted to provide heat for retorting hydrocarbon-containing fines and heating recycle retort gas. In the process, raw oil shale that has been crushed and screened in a preparation plant, not shown, is introduced into retort 10 through line 12 and feed chute 14. Normally, the shale is crushed such that the feed will not have particles greater than 6 inches in mean diameter and preferably none greater than about 3 inches. The average particle size will normally range between about is of an inch to about 2 inches in mean diameter. A feeding device located within retort housing 16 forces the shale particles upward into retort 10 at a rate which will vary considerably depending upon the size of the retort, the solids residence time desired therein and the feeding device selected for use. The feeding device may be of any suitable design such as that shown in U.S. Pat. No. 3,361,644, the disclosure of which is hereby incorporated by reference in its entirety. The feeding device, 25 however, is preferably of the design shown in U.S. patent application Ser. No. 194,133 filed on Oct. 6, 1980, the disclosure of which is hereby incorporated by reference in its entirety.

Retorting is carried out in retort 10 in a manner similar to that described in U.S. Pat. No. 3,361,644. The raw shale feed passes upwardly through retort 10 by first traversing a lower preheating zone and then an upper retorting or pyrolysis zone. Temperatures in the lower portion of the retort are sufficiently low to condense product oil vapors from the adjacent retorting zone. As the shale progresses upwardly through the retort, its temperature is gradually increased to retorting levels by contact with countercurrent eduction gases introduced into the top of the retort through line 18. The eduction gas comprises a preheated portion of the product gas produced in retort 10. Retorting temperatures will normally vary between about 800° F. and about 1100° F., and will preferably range between about 900° F. and about 1000° F. The pressure in the retort may be either subatmospheric, atmospheric or superatmospheric, but will normally range between about 5 p.s.i.a. and about 100 p.s.i.a., preferably between about 25 p.s.i.a. and about 65 p.s.i.a.

The recycle gas is introduced into the retort through line 18 at a temperature and flow rate sufficient to heat the crushed shale to retorting temperatures. The temperature of the recycle gas will normally vary between about 950° F. and about 1150° F. The flow rate of the gas will range between about 10,000 standard cubic feet per ton of feed to about 20,000 standard cubic feet per ton of feed shale. The temperature differential between the recycle gas and the shale solids at the top of the retorting zone is normally between about 10° F. and about 100° F.

As the recycle gas passes downwardly through retort 10, it continuously exchanges heat with upwardly moving oil shale. In the upper portion of the retort, hydrocarbon materials contained within the oil shale are educed therefrom by pyrolysis, thereby producing shale oil vapors and fuel gases comprising such normally uncondensable gases as methane, hydrogen and ethane. The shale oil vapors and fuel gases pass downwardly with the recycle gas into the lower portion of the retort

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wherein the cool oil shale feed condenses the shale oil vapors. Thereafter, the vapors and gases pass into a frusto-conical product disengagement zone 20. This disengagement zone comprises peripheral slots 22 through which liquid shale oil and product vapors flow 5 into surrounding product collection tank 24. The liquid shale oil is withdrawn from the collection tank through line 26 and passed downstream for further processing. The uncondensed vapors and gases are withdrawn from the collection tank into line 28. A portion of these vapors and gases is passed downstream through line 32 for processing to recover light ends oil and water, thereby producing a high BTU product gas which can be sold as a substitute natural gas after removal of small quantities of sulfur and nitrogen-containing impurities.

Retorted or pyrolyzed shale particles are removed from the top of retort 10 through chute 30 and passed through standpipe 34 into star feeder or similar device 36. The pyrolyzed solids are discharged from the star feeder through standpipe 38 into crusher or similar 20 device 40 where the solids are reduced to a top size usually no greater than about ½ inch, normally less than inch and preferably between about i and about inch. The crusher may be any suitable device for reducing the size of the pyrolyzed solids, preferably with a minimum 25 of fines production, that is fluid-tight when operated at the desired crushing pressure. Examples of crushers suitable for use in the process include toothed roll crushers, jaw crushers, cone crushers, hammer crushers and impact crushers. The crusher is operated at any 30 desired pressure that does not cause mechanical breakdown, but is normally operated at the pressure in the retort.

The crushed solids from crusher 40 are passed through standpipe 42 into fluid-tight surge vessel 44 35 where the solids are maintained in a fluidized state by an inert gas or steam introduced near the bottom of the surge vessel through line 46. The fluidizing gases are introduced at such a rate to maintain the crushed, retorted or pyrolyzed shale particles in a fluidized state 40 while the pressure in the upper region of the surge vessel is balanced so that little if any gas flows through standpipe 42 and crusher 40 either to or from the retort. The balance of pressures in the surge vessel seals the retort and prevents escape of retort gases.

The particles in surge vessel 44 are passed through standpipe 48 and slide valve 50 into lift pipe 52 where they are entrained in gas introduced into the lift through line 49. Normally, the gas introduced into the lift pipe is a mixture of air introduced into line 49 via line 56 and 50 fluidizing gas withdrawn overhead of surge vessel 44 through line 58. The gas is introduced into lift pipe 52 at a velocity and pressure sufficient to raise the crushed, pyrolyzed shale particles to the entrance of cyclone separator or similar device 60. If desired, a portion of 55 the gas supplied to lift pipe 52 through line 49 may be replaced with either steam or an inert gas.

The gas flowing upwardly in lift pipe 52 will contain primarily air but also will include hydrogen sulfide plus sulfur dioxide formed by combustion of gaseous sulfur 60 components entering the lift pipe or by combustion of sulfur-containing gases released from the pyrolyzed shale particles in the lift pipe. The gas will also contain carbon monoxide, hydrogen, and light fuel gases such as methane, ethane and the like. Some of these gases are 65 produced as the pyrolyzed shale particles pass downwardly through standpipe 38, crusher 40, standpipe 42, surge vessel 44 and standpipe 48. The fuel gases may

only be partially consumed during combustion in the lift pipe when net reducing conditions are employed. Also, fuel gases may be released from the pyrolyzed particles as they pass through the lift pipe.

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The mixture of gas and pyrolyzed shale solids enters cyclone separator 60 where the gas is removed from the solids and passed overhead of the separator through line 66 into the top of fluidized bed combustor or similar device 64. The solids removed from the gas in separator 10 60 pass downwardly through dip leg 62 into the fluidized bed in combustor 64. The fluidized bed consists of hot solids which extend upwardly within the combustor above an internal grid or similar distribution device not shown in the drawing. Pyrolyzed solids are also introduced into the fluidized bed combustor through line 68. Normally, these pyrolyzed solids will be obtained by retorting finer particles of shale or other hydrocarbon-containing solids external to the combustor as described

in more detail hereinafter. The pyrolyzed solids introduced into the fluidized bed combustor are contacted and fluidized with an oxygen-containing gas introduced into the bottom of the combustor through line 70. A sufficient amount of the oxygen-containing gas, which is preferably air, is introduced into the combustor such that the majority of the organic material in the pyrolyzed particles present in the combustor reacts exothermally with oxygen to form carbon dioxide, carbon monoxide, sulfur oxides, nitrogen oxides and decarbonized solids. The temperature in the combustor will normally be maintained between about 1250° F. and about 1700° F., preferably between about 1300° F. and about 1650° F., while the pressure is maintained at about atmospheric pressure. In general, the combustor is operated so that the maximum amount of heat energy is derived from the combustible materials introduced into the fluidized bed.

The concentration of molecular oxygen in the oxygen-containing gas introduced into combuster 64 will normally range between about 10 volume percent and about 21 volume percent. In general, the oxygen concentration is adjusted to minimize emissions of nitrogen oxides such that below 400 p.p.m.v., preferably below 300 p.p.m.v., of nitrogen oxides are produced. Normally, the combustion in combustor 64 is carried out so that no more than about 20 percent of the organic material that was originally present in the pyrolyzed shale solids removed from retort 10 through chute 30 is present. Preferably, no more than 10 percent remains, most preferably less than 5 percent.

The light hydrocarbon fuel gases introduced into the top of the fluidized bed combuster 64 through line 66 are combusted along with the organic material on the solids in the combustor to supply additional heat energy for the process of the invention. The sulfur oxides in this gas and the sulfur oxides produced within the combustor react with alkaline components of the decarbonized solids in the combustor to produce stable inorganic salts, thereby minimizing the amount of sulfur emissions in the flue gas from the combustor.

The flue gas leaving the fluidized bed in combustor 64 passes through the upper section of the combustor, which serves as a disengagement zone where particles too heavy to be entrained by the gas leaving the vessel are returned to the bed. If desired, this disengagement zone may include one or more cyclone separators or the like for the removal of relatively large particles from the gas. The gas will normally contain a mixture of carbon monoxide, carbon dioxide, unreacted oxygen,

nitrogen, entrained fines, and small quantities of sulfur oxides and nitrogen oxides. To best utilize the heat in the flue gas it is normally divided into two streams which are withdrawn separately overhead of the combustor. The first stream is removed through line 72 and 5 passed into steam generator or similar heat exchange vessel 74 where it passes in indirect heat exchange with boiler water introduced into the heat exchanger through line 76. Steam is removed, from vessel 74 through line 78 and passed downstream for subsequent 10 use. Fine particulates which are removed from the flue gas in steam generator 74 exit the vessel through line 80 while the remainder of the gas and entrained fines pass through line 82 into cyclone separator or similar device 84. Here additional fine particulates are removed from 15 the gas through dip leg 86. The gas taken overhead of the separator through line 88 is passed downstream through lines 90 and 92 to a bag house or scrubbing system to remove the last traces of fines before the gas is discharged into the atmosphere.

The second stream of flue gas removed from fluid bed combuster 64 is withdrawn overhead through line 94 and passed into recycle gas preheater or similar heat exchange vessel 96 where the flue gas is passed in indirect heat exchange with a portion of the product gas 25 removed from retort 10 through line 28 and passed into recycle gas preheater 96 via line 98. The recycle product gas is heated from about 200° F. to between about ::650° F. and about 950° F. in the recycle gas preheater and is removed from the preheater through line 100. 30 Fine particulates which drop out of the flue gas in the preheater are removed from the bottom of the vessel through line 102 while the cooled flue gas and remaining fine particulates are withdrawn from the preheater through line 104 and passed into cyclone separator or 35 similar device 106 where additional fines are removed through dip leg 108. The cool flue gas containing fine particulates is removed overhead from the separator through line 110 and is mixed with gases in lines 178, 88 and 132 prior to being sent to a bag house or scrubbing 40 system to remove the last traces of fines prior to discharge into the atmosphere.

The fines exiting recycle gas preheater 96 through line 102 are combined with the fine particulates removed from cyclone separator 106 through dip leg 108, 45 the fine particulates removed from heat exchange vessel 74 through line 80 and the fines removed from cyclone separator 84 through dip leg 86. The combined mixture of all these particles is passed through line 102 into fines cooler or similar vessel 114. Here the solids are cooled 50 by contact with water which is introduced into vessel 114 via line 118 and distribution means 116. Air is introduced into fines cooler 114 through line 120 at a rate sufficient to fluidize the fines within the vessel. The amount of water introduced into the fines cooler is 55 regulated such that all the water is vaporized by the heat content of the fines. Decarbonized shale fines essentially free of moisture are removed from the cooler through line 122 and transported to a disposal site through line 124.

A mixture of air, vaporized water and entrained fines is removed overhead of fines cooler 114 through line 126 and passed to cyclone separator or similar device 128. Here the fines are removed from the mixture of dip leg 130 into line 124 for transportation to the disposal site. The mixture of water vapor and air is removed overhead of cyclone separator 128 through line

132, mixed with the flue gas in line 90 and passed through line 92 to a bag house or other treatment facility to remove fines.

In a typical prior art process as described above wherein oil shale is retorted by passing it upwardly in countercurrent contact with a downflowing pyrolysis or eduction gas, fine particles in the feed shale tend to fill void spaces between the larger particles during retorting. This increases pressure drop through the retort and may result in maldistribution of the pyrolysis gases as they pass through the bed of shale particles undergoing retorting. Also, a large recycle gas preheater is typically required to heat the recycle pyrolysis gas to a sufficient temperature to effect the pyrolysis in the retort. A preheater of the size required is expensive and the high temperatures necessary in the preheater may result in coking of the heat exchange tubes therein.

It has now been found that large pressure drops in the retort can be avoided by removing fines from the feed material to the retort and separately retorting them in a second retorting zone by contacting the fines with hot. decarbonized solids produced by combusting the retorted shale from the primary retort. In addition, the gas from the recycle gas preheater can be passed into the secondary retorting zone wherein heat from the hot, decarbonized solids also serves to further heat the recycle gas. By supplying heat to the recycle gas in this manner, the temperature at which the recycle gas preheater is operated can be decreased along with the size of the preheater, thereby minimizing coking of the preheater's heat exchange tubes.

Referring again to the drawing, a portion of the hot, decarbonized solids produced in fluidized bed combustor 64 is passed downwardly through transfer line 134 and slide valve 136 into fluidized bed retort 138 where the solids are contacted with hydrocarbon-containing fines introduced into fluidized bed retort 138 through line 140. Both the decarbonized solids and the hydrocarbon-containing fines are maintained in the fluidized state in retort 138 by preheated recycle product gas removed from recycle gas preheater 96 through line 100 and introduced into the bottom of retort 138. The heat from the hot, decarbonized solids produced in combustor 64 is directly transferred to the hydrocarbon-containing fines and the recycle gas in the retort. The heat pyrolyzes hydrocarbon material in the the fines, thereby producing pyrolysis products containing gases and vapors. The recycle gas is further heated to retorting temperatures similar to those required in retort 10.

The fines introduced into retort 138 through line 140 may be raw fines containing organic material that are produced from any hydrocarbon-containing solids. Examples of such fines include fines derived from oil shale, coal, lignite, solid organic wastes, tar sands, petroleum coke and the like. In general, the largest fines will have a 1 inch top size, preferably a 1 inch top size. Normally, the fines will be oil shale fines produced during the mining of the oil shale fed to retort 10. Typically, the amount of fines introduced into retort 138 will be be-60 tween about 4 and about 10 weight percent of the shale fed into retort 10 through line 12.

In general, sufficient hot, decarbonized solids are passed into fluidized bed retort 138 from combustor 64 such that the temperature of the solids and gases in the water vapor and air and passed downwardly through 65 retort will range between about 900° F. and about 1200° F., preferably between about 950° F. and about 1150° F. Normally, between about 1.5 and about 4 pounds of hot solids are introduced in the retort for every pound of hydrocarbon-containing fines. The solids residence time in the retort will range between about 2 minutes and about 45 minutes, preferably between about 5 minutes and about 30 minutes. Normally, no air or molecular oxygen is introduced into retort 138 so that the pyrolysis of the hydrocarbon-containing fines and the heating of the recycle gas are carried out in the substantial absence of molecular oxygen. The presence of oxygen in the retort would result in decreased yields of lower quality product from the overall process.

A large majority of hydrogen sulfide and carbon dioxide formed in retort 138 or which is present in the recycle gas introduced into the retort through line 100 will be absorbed by inorganic constituents in the solids undergoing retorting. Normally, the sulfur compounds 15 will react with calcium and magnesium components to form calcium and magnesium sulfide. Heated gas substantially free of hydrogen sulfide and carbon dioxide and containing fine particulates is removed from retort 138 overhead through line 142 and passed to cyclone 20 separator or similar device 144 where the fine particulates are removed and returned to the retort through dip leg 146. The remaining hot gas, normally at a temperature of between about 950° F. and about 1150° F. and substantially free of particulates, is removed overhead 25 from cyclone separator 144 through line 18 and recycled to retort 10 where it serves to supply the heat necessary to educe hydrocarbons from the feed shale introduced into the retort through line 12 and feed

The pyrolyzed fines produced in fluidized bed retort 138 by retorting the hydrocarbon-containing fines will contain organic material in the form of coke. In order to utilize this organic material to supply additional heat for the process, the fines are passed from the retort to fluid- 35 ized bed combustor 64. The pyrolyzed fines are removed in intimate mixture with the decarbonized solids passed into the retort through transfer line 134 from the bottom of the retort through transer line 148. The mixture of solids is then passed through valve 150 into lift 40 pipe 68 where the solids are entrained in a stream of air introduced into the lift pipe via line 152. The air carries the mixture of pyrolyzed fines and decarbonized solids through lift pipe 68 into fluidized bed combustor 64 where the coke on the fines is burned. Any calcium and 45 magnesium sulfides formed by absorption of hydrogen sulfide in retort 138 are partially oxidized in the fluidized bed combustor and thereby converted into sulfites and sulfates. The amount of solids circulating from retort 138 into fluidized bed combustor 64 can be ad- 50 justed to control the temperature in the combustor.

In some cases, additional fuel may be required for the fluidized bed combustor either during startup or normal operations. If this is the case, the additional fuel in the form of fuel gas, fuel oil, raw oil shale fines, coal fines or 55 any combinations thereof may be introduced into the combustor through line 154.

In general, the heat needed in fluidized bed retort 138 will require only that a portion of the hot, decarbonized solids in fluidized bed combustor 64 be passed through 60 transfer line 134 and valve 136 into the retort. The remaining portion of the decarbonized solids is withdrawn from the bottom of combustor 64 and passed through transfer line 156 and slide valve 158 into cooling vessel 160. Here the decarbonized solids are fluidized with air at ambient temperature introduced into the bottom of vessel 160 through line 162. In the upper portion of the cooling vessel, the hot fluidized solids

supply heat indirectly to water introduced into the vessel through line 164 to produce steam which is withdrawn from the vessel through line 166. In the lower portion of cooling vessel 160, boiler feed water is passed into the vessel through line 168 and heated by indirect heat exchange with the decarbonized solids. Preheated boiler water is removed from the lower portion of the vessel through line 170. As a result of this heat recovery, the temperature of the decarbonized shale solids 10 drops from that in the combustor, normally between about 1300° F. and about 1650° F., to between about 300° F. and about 450° F. The residence time in vessel 160 will normally be sufficient to accomplish the above temperature drop while allowing for combustion of some or all of the residual organic material remaining in the decarbonized solids. Normally, the residence time will be between about 20 minutes and about 40 minutes.

The cooled, decarbonized shale solids exit the bottom of cooling vessel 160 through transfer line 172 and valve 174. The cooled solids, which are essentially moisture-free ash, are passed through line 176 and combined with the cooled shale fines in lines 122 and 130 to form a mixture which is sent to a disposal site via line 124. A conventional system for controlling wetting, not shown in the drawing, may form a part of the disposal system. For example, the decarbonized shale particles in line 124 may be sent to a pug mill and therein mixed with water to form a cement type mixture.

Air containing fine particulates is removed overhead of cooling vessel 160 through line 178, combined with flue gas in lines 110, 88 and 132 to form a mixture of gases which is passed through line 90 to a bag house or scrubbing system. Here the fines are removed from the mixture of gases, which is then discharged into the atmosphere.

It will be apparent from the foregoing that the process of the invention provides a method in which raw oil shale fines can be retorted independently of the main retort where the larger shale particles undergo pyrolysis. Because of this, fine particles do not tend to plug the shale bed in the main retort, thereby increasing pressure drop. The vapors generated by the retorting of the shale fines external to the main retort can be added to the volume of recycle gas which in turn will reduce horsepower requirements of the recycle compressor. A large majority of hydrogen sulfide and carbon dioxide in the recycle gas exiting the recycle gas preheater is removed in the vessel in which the fines are pyrolyzed and therefore downstream requirements for product gas clean up are minimized. Coke produced during the retorting of the raw fines is also burned in the combustor to provide additional heat efficiencies for the process. Also, the hot solids produced in the combustor are used to heat recycle gas and therefore the recycle gas preheater requires much less heat exchange surface and lower temperatures. This, in turn, results in less coking of the heat exchange tubes in the preheater.

Although this invention has been primarily described in conjunction with a preferred embodiment, it is evident that many alternatives, modifications, and variations will be apparent to those skilled in the art in light of the foregoing description. Accordingly, it is intended to embrace within the invention all such alternatives, modifications and variations that fall within the spirit and scope of the appended claims.

We claim:

1. A process for retorting oil shale solids which comprises:

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(a) contacting said oil shale solids with a hot, non-oxidizing recycle gas comprising gases and vapors from step (e) under pyrolysis conditions in a first retorting zone in the substantial absence of the hot decarbonized solids formed in step (c) to produce an effluent containing pyrolysis products and pyrolyzed oil shale solids:

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- (b) recovering liquid and gaseous hydrocarbons from the effluent of said first retorting zone;
- (c) contacting said pyrolyzed oil shale solids with a gas 10 containing molecular oxygen in a combustion zone under conditions such that at least a portion of the organic material remaining in said solids is burned to produce hot, decarbonized solids and a hot flue gas;
- (d) contacting at least a portion of the gases in the effluent from said first retorting zone in the substantial absence of molecular oxygen with a portion of said hot, decarbonized solids produced in step (c) in the presence of added hydrocarbon-containing fines in a second retorting zone such that said gases are heated and said fines are pyrolyzed to produce pyrolyzed fines and pyrolysis products containing gases and vapors: and
- (e) recovering gases and vapors from said second retorting zone.
- 2. A process as defined by claim 1 including the additional step of passing said portion of gases in the effluent from said first retorting zone in indirect heat exchange with said hot flue gas produced in step (c) to preheat said portion of gases prior to contacting said portion of gases with said portion of hot, decarbonized solids produced in step (c).
- 3. A process as defined by claim 1 wherein said gas containing molecular oxygen comprises air.
- 4. A process as defined by claim 1 wherein said hydrocarbon-containing fines comprises oil shale fines.
- 5. A process as defined by claim 1 wherein said first retorting zone is maintained at a temperature between about 800° F. and about 1100° F.
- 6. A process as defined by claim 1 wherein the temperature in said second retorting zone is maintained between about 900° F. and about 1200° F.
- 7. A process as defined by claim 1 wherein the temperature in said combustion zone is maintained between 45 (d) passing at least a portion of the gases in the effluent about 1250° F. and about 1700° F.
- 8. A process as defined by claim 1 wherein said pyrolyzed fines produced in said second retorting zone are passed to said combustion zone.
- 9. A process as defined by claim 1 wherein said combustion zone and said second retorting zone contain fluidized beds.
- 10. A process as defined by claim 1 wherein said oil shale solids are contacted in said first retorting zone with a downflowing stream of said recycle gas as said 55 solids move upward through said retorting zone.
- 11. A process for retorting oil shale solids which comprises:
- (a) contacting said oil shale solids with a hot, non-oxidizing recycle gas under pyrolysis conditions in a first force retorting zone to produce an effluent containing pyrolysis products and pyrolyzed oil shale solids;
- (b) recovering liquid and gaseous hydrocarbons from the effluent of said first retorting zone;
- (c) contacting said pyrolyzed oil shale solids with air in 65 a fluidized bed combustion zone external to said first retorting zone under conditions such that at least a portion of the organic material remaining in said

- 12 solids is burned to produce hot, decarbonized solids and a hot flue gas;
- (d) passing at least a portion of the gases in the effluent from said first retorting zone in indirect heat exchange with at least a portion of said hot flue gas produced in step (c) to preheat said gases;
- (e) contacting said preheated gases from step (d) in the substantial absence of molecular oxygen with a portion of the hot, decarbonized solids produced in step (c) in the presence of added oil shale fines in a second retorting zone such that said preheated gases are further heated and said fines are pyrolyzed to produce pyrolyzed fines and pyrolysis products containing gases and vapors; and
- (f) using the gases and vapors in the effluent from said second retorting zone as said recycle gas in step (a).
- 12. A process as defined by claim 11 wherein said second retorting zone is a fluidized bed retorting zone.
- 13. A process as defined by claim 11 wherein said pyrolyzed fines produced in said second retorting zone are passed to said fluidized bed combustion zone.
- 14. A process as defined by claim 1 wherein said first retorting zone is contained within a first vessel and said second retorting zone is contained within a second vessel separate from said first vessel.
- 15. A process as defined by claim 11 wherein said first retorting zone is contained within a first vessel and said second retorting zone is contained within a second vessel separate from said first vessel.
- 16. A process for retorting oil shale solids which comprises:
- (a) contacting said oil shale solids with a hot, non-oxidizing recycle gas comprising gases and vapors from step (f) under pyrolysis conditions in a first retorting zone to produce an effluent containing pyrolysis products and pyrolyzed oil shale solids;
- (b) recovering liquid and gaseous hydrocarbons from the effluent of said first retorting zone;
- 40 (c) contacting said pyrolyzed oil shale solids with a gas containing molecular oxygen in a combustion zone under conditions such that at least a portion of the organic material remaining in said solids is burned to produce hot, decarbonized solids and a hot flue gas;
 - (d) passing at least a portion of the gases in the effluent from said first retorting zone in indirect heat exchange with said hot flue gas produced in step (c) to preheat said gases;
 - (e) contacting said preheated gases from step (d) in the substantial absence of molecular oxygen with a portion of the hot, decarbonized solids produced in step (c) in the presence of added hydrocarbon-containing fines in a second retorting zone such that said preheated gases are further heated and said fines are pyrolyzed to produce pyrolyzed fines and pyrolysis products containing gases and vapors; and
 - (f) recovering gases and vapors from said second retorting zone.
 - 17. A process for retorting oil shale solids which comprises:
 - (a) passing said oil shale solids upward through a first retorting zone in contact with a downflowing hot, non-oxidizing recycle gas comprising gases and vapors from step (e) under pyrolysis conditions to produce an effluent containing pyrolysis products and pyrolyzed oil shale solids;
 - (b) recovering liquid and gaseous hydrocarbons from the effluent of said first retorting zone;

(c) contacting said pyrolyzed oil shale solids with a gas containing molecular oxygen in a combustion zone under conditions such that at least a portion of the organic material remaining in said solids is burned to produce hot, decarbonized solids and a hot flue gas;
(d) contacting at least a portion of the gases in the effluent from said first retorting zone in the substantial absence of molecular oxygen with a portion of said hot, decarbonized solids produced in step (c) in the

presence of added hydrocarbon-containing fines in a second retorting zone such that said gases are heated and said fines are pyrolyzed to produce pyrolyzed fines and pyrolysis products containing gases and vapors; and

(e) recovering gases and vapors from said second retorting zone.

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UNITED STATES PATENT AND TRADEMARK OFFICE CERTIFICATE OF CORRECTION

PATENT NO. :

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DATED

07/23/85

INVENTOR(S):

Deering, Gowdy and Kelley

It is certified that error appears in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

Col. 11, claim 3, line 34; claim 4, line 36; claim 5, line 38; claim 6, line 41; claim 7, line 44; claim 8, line 47; and claim 9, line 50, after "claim" delete "1" and insert therefor --14--.

Signed and Sealed this Tenth Day of November, 1987

Attest:

DONALD J. QUIGG

Attesting Officer

Commissioner of Patents and Trademarks



United States Patent [19]

Nicaud et al.

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[54] CATALYZED DEGRADATION OF THERMOPLASTIC POLYOLEFINS AND INCORPORATION OF THE DEGRADATION PRODUCTS INTO HEAVY HYDROCARBONS

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[52] U.S. Cl. 521/47; 521/40.5; 524/62; 528/485; 528/490; 523/351

525/54.5, 333.7, 370, 371; 585/241; 524/62; 523/351; 528/485, 490 [56] References Cited

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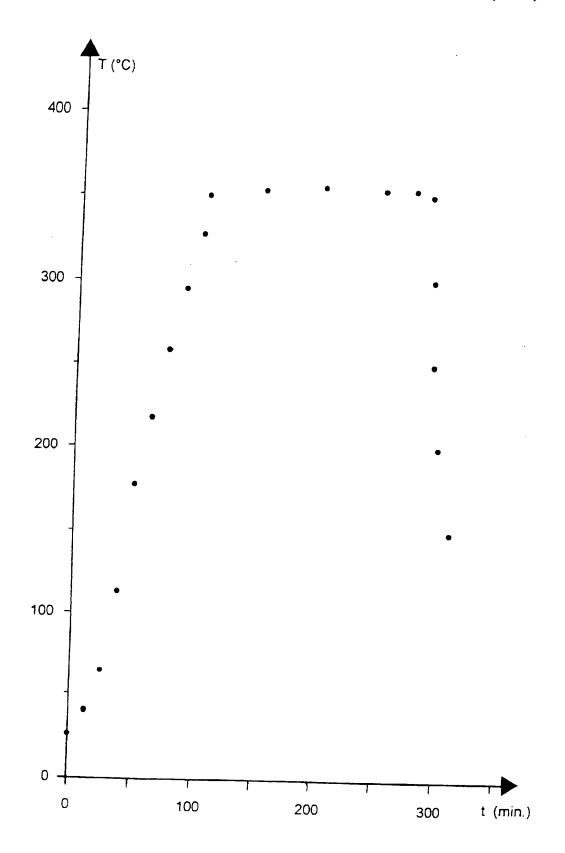
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ABSTRACT

A process for degrading thermoplastic polyolefins, in particular high or medium density polyethylenes, uses as a catalyst a mineral solid containing copper or a mineral solid containing aluminium and silicon and with an acid nature, at a temperature of 200° C. to 450° C., degradation being accompanied by incorporation of the degradation products into a heavy hydrocarbon such as a bitumen base.

Incorporating degraded thermoplastic polyolefins, in particular degraded high or medium density polyethylenes, into heavy hydrocarbons can produce compositions with industrial and road applications.

20 Claims, 1 Drawing Sheet



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CATALYZED DEGRADATION OF THERMOPLASTIC POLYOLEFINS AND INCORPORATION OF THE DEGRADATION PRODUCTS INTO HEAVY HYDROCARBONS

The invention relates to a process for degrading thermoplastic polyolefins, in particular high or medium density polyethylenes, and incorporating the degraded thermoplastic polyolefins into heavy hydrocarbons.

It also relates to upgrading thermoplastic polyolefin waste by incorporating their degradation products into heavy 10

hydrocarbons such as bitumen bases.

Plastic packaging constituted by polyolefins must be recycled for environmental protection reasons. This is particularly the case for high density polyethylene films and containers used for packaging lubricating oils, household detergents, phytosanitary products, etc.; such products constitute well identified sources of high density polyethylene for recycling.

Incorporating polyolefin waste into various heavy hydrocarbons such as bitumen bases to produce modified bitumen compositions with properties which may be improved has been considered, particularly for industrial and road applications

However, the thermal and/or mechanical degradation methods which have been described are principally applicable to low density polyethylenes and cannot incorporate 25 large quantities of all types of thermoplastic polyelefins, in particular all types of high or medium density polyethylenes, into heavy hydrocarbons, particularly when such polyethylenes are mixtures, as is often the case for polyethylenes recovered for recycling.

We have now, unexpectedly, discovered that certain solid mineral compounds could be advantageously used to degrade thermoplastic polyolefins, in particular when they are as a mixture. These compounds can increase the degradation rate of all types of polyolefins and thus can reduce the time required to degrade the polyolefin(s).

The invention thus proposes a process for degrading thermoplastic polyolefins which comprises mixing, at high temperature, at least one thermoplastic polyolefin with at least one catalyst consisting essentially of a solid mineral compound containing copper or a solid mineral compound containing aluminium and silicon and having an acid nature, in the presence of at least one heavy hydrocarbon.

The degraded thermoplastic polyolefins are thus upgraded in accordance with the invention by incorporating them into a heavy hydrocarbon (or a mixture of heavy 45 hydrocarbons) such as a bitumen base.

The degraded thermoplastic polyolefins of the invention can also be upgraded into a variety of other hydrocarbon compositions such as polyethylene waxes or paraffinic hydrocarbons such as paraffin waxes.

The thermoplastic polyolefins to which the degradation process of the invention applies are generally homopolymers and copolymers of α -olefins containing 2 to 8 carbon atoms, more particularly high or medium density polyethylenes, polypropylenes or ethylene-propylene copolymers, these thermoplastic polyolefins generally being more than 35% crystalline. As an indication, high density polyethylenes (partially crystalline) melt in the region of about 135° C. and polypropylenes (partially crystalline) melt in the region of about 165° C.

Particular examples of high density polyethylenes are 60 polyethylenes with high mass average molecular weights, for example about 150,000 to 200,000, and very high molecular weight polyethylenes, for example up to about 500,000 to 1,000,000.

The polyolefins considered in the invention can also 65 consist of copolymers of ethylene or propylene with 1-butene, 1-hexene or 1-octene.

Mixtures of two or more thermoplastic polyolefins can also be considered.

The heavy hydrocarbons which can be used in the invention to obtain bitumen compositions can be mixtures of heavy hydrocarbons hereinafter termed "conventionally obtained bitumens", to differentiate them from "synthetic bitumens" described below. These conventionally obtained bitumens originate from crude oil, bituminous schists, heavy oils, bituminous sand, etc., or from coal.

These heavy hydrocarbons are, for example, characterized by a softening temperature, measured using the standard NF-T-66 008 method (ball-ring temperature) of at least 10° C., and/or a kinematic viscosity, measured at 100° C., of about 50 to about 700 mm²/s.

The heavy hydrocarbons considered in the invention can, for example, consist of:

- a) the heaviest fraction obtained by direct distillation of crude oil at atmospheric pressure or under reduced pressure;
- b) the heavy phase obtained by solvent deasphalting a heavy fraction obtained using the processes described in a);
- c) the oxidation product, in the presence or otherwise of catalysts, of a heavy fraction from a) or a heavy phase from b);
- d) the oxidation product, in the presence or otherwise of catalysts, of a mixture: of a heavy fraction from a) or a heavy fraction from b) and

a distillate, or

- an aromatic extract obtained from dearomatisation of lubricating oils, or
- a deasphalting pitch; or
- e) a mixture of an oxidised product obtained from b) or c), and

a distillate, or

- an aromatic extract obtained from dearomatisation of lubricating oils, or
- a deasphalting pitch, or
- a heavy fraction from a) or a heavy phase from b).

The mixture of heavy hydrocarbons which can be used can also be a synthetic bitumen with characteristics which are close to those of a conventionally obtained bitumen as described above: it could thus, for example, be a clear synthetic binder which can be coloured by adding pigments.

The heavy hydrocarbon mixtures can also consist, for example, of petroleum resins or indene-coumarone resins, for example mixed with aromatic and/or paraffinic hydrocarbons.

The petroleum resins can be prepared by polymerisation of unsaturated hydrocarbons present in unsaturated petroleum fractions such as the fractions obtained by thermal cracking or steam cracking or by pyrolysis.

Indene-coumarone resins are obtained from coal tar.

The thermoplastic polyolefin degradation process of the invention can more particularly be defined as comprising mixing, for example by mixing at a temperature of 200° C. to 450° C., preferably 250° C. to 375° C., of at least one thermoplastic polyolefin in the presence of a proportion, for example 0.1% to 6% by weight with respect to the weight of the thermoplastic polyolefin, of at least one catalyst selected from mineral solids containing copper or those containing aluminium and silicon and having an acid nature, in the presence of at least one heavy hydrocarbon.

Examples of solid mineral compounds containing copper or those containing aluminium and silicon and with an acid nature which can be used as catalysts in the degradation process of the invention are clays activated by an acidic treatment, acid silica-aluminas or acid zeolites, or cuprous oxide Cu₂O. Cuprous oxide is preferred and can in particular be used in a proportion of 0.1% to 2% by weight.

Degrading the thermoplastic polyolefin and incorporating it into the heavy hydrocarbon mixture is carried out by mixing, in the presence of a catalyst as defined above, a proportion of 5% to 99% by weight of a mixture of heavy hydrocarbons with a proportion of 1% to 95% by weight 5 (including catalyst) of thermoplastic polyolefin.

Thus the degraded thermoplastic polyolefin(s) can be incorporated into the heavy hydrocarbons under consideration in proportions which are suitable for the intended applications for the bitumen compositions, i.e., generally about 1% to 25% by weight of degraded thermoplastic polyolefin (this proportion including the catalyst) with 75% to 99% by weight of heavy hydrocarbon mixture for industrial applications (for example the production of roofing) or road applications (pavement coatings). More particularly, a proportion of about 1% to 15% by weight of degraded 15 thermoplastic polyolefin for road applications and about 3% to 25% by weight for industrial applications is incorporated into the heavy hydrocarbons.

More particularly, the simultaneous degradation and incorporation process of the invention means that "bitumen- 20 polymer' compositions can be directly obtained in all proportions, for example in proportions suitable for industrial and road applications, these proportions being, as indicated above, about 1% to 15% by weight for road applications and about 3% to 25% by weight for industrial 25 applications.

It may be advantageous to prepare "bitumen-polymer" mixtures which are concentrated in degraded polyolefins, known as "master mixtures", containing, for example, a proportion of 40% to 60% by weight of bitumen and a proportion of 60% to 40% by weight of degraded polyolefin.

These master mixtures can easily be stored. They can subsequently be diluted in bitumen for use to adjust the concentration of degraded polyolefin to a value which is suitable for the envisaged application.

When the cold behaviour of bituminous compositions 35 prepared using the invention is to be improved, elastomers can be incorporated into them in the usual proportions, for example SBS (styrene-butadiene-styrene) rubbers or atactic polypropylenes.

The mixing operations carried out in the process of the 40 invention can be effected using different types of reactors and mixers when carried out batchwise; they can also be carried out continuously, for example in a single or twinscrew extruder.

obtained using the process of the invention can, for example, have a penetrability of 20 to 330 at 25° C., measured for 1/10 mm,; they may have a dynamic viscosity of 18 to 440 Pa.s., measured at 60° C., and a kinematic viscosity of 100 to 530

The bitumen compositions obtained can also have a 50 penetrability of 70 to 360 at 15° C., for 1/10 mm, and in this case a dynamic viscosity of 4.5 to 18 Pa.s., measured at 60° C., and a kinematic viscosity, measured at 135° C., of 50 to $100 \text{ mm}^2/\text{s}$.

The following examples illustrate the invention.

EXAMPLES 1 TO 6

Bitumen, which had been heated to 80° C. to allow it to flow, was charged into an electrically heated reactor provided with an anchor stirrer and inert gas circulation.

In the examples, a bitumen base of petroleum origin with the following characteristics was used:

kinematic viscosity at 100° C.: 615 mm²/s;

penetrability at 25° C. for 1/10 mm: 500; (using NF-T-66

softening point: (ball-ring temperature 24.5° C. using NF-T-66 008)

In Table 1, this bitumen is shown as Base B.

In each of Examples 1 to 4, a proportion of 11.5% by weight with respect to the total weight (bitumen+ polyethylene) of a high density polyethylene recovered from used oil containers round to a coarse powder with a smallest dimension of the order of a few millimeters was introduced into the bitumen. This polyethylene is termed "recycled" in

The containers were constituted by high density polyethylene with the following characteristics: a density of 0.949, a melt flow index of 0.5 g/10 min, measured at 190° C. at a load of 2.16 kg using ASTM standard D 1238, and molar masses, measured using gel permeation chromatography, of 150000 for the mass average and 20000 for the number average. The crystallinity, measured using differential scanning calorimetry (DSC), was of the order of 50%. Before transformation into containers, it had been sold by BASF under the trade name "Lupolen 5021D®".

In Examples 5 and 6, the same polyethylene as provided in granular form by the manufacturer was used, i.e., before transformation into containers ("virgin" in Table 1).

The catalyst used in Example 3 was a montmorillonite based clay (hydrated aluminium silicate with a SiO₂/Al₂O₃ ratio of 4/1) treated with a mineral acid. It is sold by Sud Chemie under the trade name "Tonsil Optimum FF®". It is hereinaster termed "Tonsil®". This catalyst was used in a proportion of 0.5% by weight with respect to the bitumen+ polyethylene ensemble (i.e., about 4.4% with respect to the polyethylene). The catalyst used in Examples 4 and 6 was cuprous oxide (Cu₂O). It was used in a proportion of 0.1% by weight with respect to the bitumen+polyethylene ensemble (i.e., about 0.87% with respect to the polyethylene). A catalyst was not used in Examples 1, 2 and

In each case, the mixture was heated to 350° C. with stirring (rotation rate: 350 revolutions per minute), the temperature rise period being about 2 hours and the time during which the temperature was held being, depending on the case, 1 hour or 3 hours, as indicated in Table 1. FIG. 1 shows the type of temperature profile obtained when the temperature is held at 350° C. for 3 hours.

After the temperature maintenance phase, the mixture was cooled in the reactor to a temperature of the order of 200° C., then cast through a bottom flap into plates a few millimeters thick, and the appearance was evaluated.

The result of the observations is shown in Table 1. Examples 1, 2 and 5 in Table 1 show that in the absence of Depending on its final use, the bitumen compositions 45 catalyst, simple heat treatment of the bitumen-polyethylene mixture at 350° C. for 1 hour did not produce a satisfactory dispersion of the "virgin" or "recycled" polyethylene (even after 3 hours in the latter case) in the bitumen. The appearance of the cast plates was very irregular with heterogeneities which were visible to the naked eye, which meant that the dispersion was poor. In contrast, cast plates of all of the mixtures formed in the presence of the catalysts of the invention (Examples 3, 4 and 6) had a smooth appearance which meant that dispersion of the polyethylene in the bitumen was very good. The heterogeneous structure of the 55 latter mixtures could not be seen with the naked eye and required the use of an optical microscope with an epifluorescent lamp to reveal the heterogeneous structure on a microscopic scale.

> The preceding examples can be repeated with similar 60 success by substituting the generically or specifically described reactants and/or operating conditions of this invention for those used in the preceding examples.

> From the foregoing description, one skilled in the art can easily ascertain the essential characteristics of this invention 65 and, without departing from the spirit and scope therof, can make various changes and modifications of the invention to adapt it to various usages and conditions.

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TABLE 1

| | Example | 1 | 2 | 3 | 4 | 5 | 6 |
|---------------|-----------------------------|---------------|---------------|-------------|-------------------|---------------|-------------------|
| Simultaneous | Polyethylene | "recycled" | "recycled" | "recycled" | "recycled" | "virgin" | "virgin" |
| degradation | wt % | 11.5 | 11.5 | 11.5 | 11.5 | 11.5 | 11.5 |
| and | Bitumen | Base B | Base B | Base B | Base B | Base B | Base B |
| incorporation | wi % | 88.5 | 88.5 | 88.5 | 88.5 | 88.5 | 88.5 |
| | Catalyst | | _ | Tonsil ® | Cu ₂ O | | Cu ₂ O |
| | wt % | | _ | 0.5 | 0.1 | _ | 0.1 |
| | Temperature (° C.) | 350 | 350 | 350 | 350 | 350 | 350 |
| | Time (hours) | 1 | 3 | 1 | 1 | 1 | 1 |
| | Plate appearance 190° C. | heterogeneous | heterogeneous | homogeneous | homogeneous | heterogeneous | homogeneou |

The entire disclosure of all applications, patents and publications, cited above and below, and of corresponding French application No. 97/05.050, are hereby incorporated by reference.

What is claimed is:

- 1. A process for degrading a thermoplastic polyolefin and incorporating it into a mixture of heavy hydrocarbons, comprising
 - mixing, at a temperature of 200° C. to 450° C., at least one thermoplastic polyolefin in the presence of at least one catalyst selected from the group consisting of mineral solids containing copper and mineral solids containing aluminum and silicon and having an acid nature, with at least one heavy hydrocarbon mixture under conditions sufficient to obtain a dispersion of said thermoplastic polyolefin in said hydrocarbon mixture, said dispersion appearing homogeneous to the naked eye, but appearing heterogeneous through an optical microscope with an epifluorescent lamp.
- 2. A process according to claim 1, characterized in that the 35 proportion of thermoplastic polyolefin used is 1% to 95% by weight, catalysts included, with a proportion of 99% to 5% by weight of a mixture of heavy hydrocarbons.
- 3. A process according to claim 1, characterized in that said thermoplastic polyolefin is a homopolymer or copolymer of at least one α -olefin containing 2 to 8 carbon atoms.
- 4. A process according to claim 2, characterized in that said thermoplastic polyolefin is a high or medium density polyethylene.
- 5. A process according to claim 1, characterized in that said mixture of heavy hydrocarbons has a softening temperature, measured by the standard NF-T-66 008 method (ball-ring temperature) of at least 10° C. and/or a kinematic viscosity, measured at 100° C., of about 50 to 700 mm²/s.
- 6. A process according to claim 1, characterized in that the 50 catalyst is in a proportion of 0.1% to 6% by weight with respect to the weight of the thermoplastic polyolefin.
- 7. A process according to claim 1, characterized in that the catalyst is a at least one clay activated by at least one acid treatment, b an acid silica-alumina or c an acid zeolite.
- 8. A process according to claim 1, characterized in that the catalyst is cuprous oxide.

- 9. Aprocess according to claim 8, characterized in that the proportion of cuprous oxide is 0.1% to 2% by weight with respect to the weight of the thermoplastic polyolefin.
- 10. A process according to claim 1, characterized in that the temperature is 250° C. to 375° C.
- 11. A process according to claim 1, characterized in that said mixture of heavy hydrocarbons consists essentially of bitumen.
- 12. A process according to claim 11, characterized in that for road applications, the thermoplastic polyolefin is incorporated into the bitumen in a proportion of 1% to 15% by weight.
- 13. A process according to claim 11, characterized in that of industrial applications, the thermoplastic polyolefin is incorporated into the bitumen in a proportion of 3% to 25% by weight.
 - 14. A process according to claim 11, characterized in that the proportion of thermoplastic polyolefin provides a master mixture containing 40% to 60% by weight of degraded thermoplastic polyolefin with 60% to 40% by weight of bitumen.
 - 15. A process according to claim 14, further comprising diluting the master mixture in bitumen to adjust the concentration of degraded thermoplastic polyolefin (catalyst included) to a value of from 1% to 25%.
 - 16. A process according to claim 1, wherein the thermoplastic polyolefin comprises recycled polyethylene oil containers.
 - 17. A process according to claim 15, wherein the concentration of degraded thermoplastic polyolefin is adjusted to a value of from 1% to 15%.
- 18. A process according to claim 15, wherein the concentration of degraded thermoplastic polyolefin is adjusted to a value of from 3% to 25%.
- 19. A process according to claim 1, wherein the mixture of heavy hydrocarbons comprises bitumen.
- 20. A process according to claim 19, wherein the mixture
 of heavy hydrocarbons further comprises an elastomer.

* * * * *

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of:

Craig N. Eatough, et al.

Docket:

8333

Serial No.: 09/954,603

Art Unit:

1764

Filed:

September 17, 2001

Examiner: Alexa A. Doroshenk

For:

CLEAN PRODUCTION OF COKE

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

Sir:

Responsive to the Office Action mailed 25 August 2003, please amend the above-identified application as follows:

IN THE CLAIMS:

Please amend the claims as indicated below:

-- 32. (currently amended) A method of producing high grade coke from low grade material without causing a pollution problem, comprising the acts of:

introducing displacing a mixture of unwashed non-elutriated low grade non-coking inexpensive coal fines and another type of inexpensive carbonaceous fines comprised of unwashed non-elutriated waste coke fine without elutriation of the fines, as a feedstock influent into a pyrolyzer;

pyrolyzing the <u>displaced</u> mixture in the pyrolyzer;

discharging coke and pyrolytic by-products as effluents from the pyrolyzer. - -

- - 33. (previously presented) A method according to Claim 32 further comprising the acts of:

feeding back tar effluent by-product from the pyrolyzer to the feedstock influent mixture; feeding back combustible off-gas effluent by-product from the pyrolyzer to the pyrolyzer and using it as a source of fuel in the pyrolyzer. - -

- - 34. (currently amended) A method according to Claim 32 further comprising the act of obtaining a mixture comprising unwashed waste coal fines and unwashed waste coke fines prior to the introducing act. -
- - 35. (previously presented) A method according to Claim 32 further comprising the act of crushing low grade coal and/or the carbonaceous waste coke prior to the introducing act, to obtain the fines. -

- - 36. (previously presented) A method according to Claim 32 further comprising the act of forming the mixture into solid objects prior to the introducing act. -
- -- 37. (previously presented) A method according to Claim 35 wherein the discharging act comprises discharging the coke as solid objects. --
- -- 38. (previously presented) A method according to Claim 33 wherein the first feeding act comprises combining the feedback tar, a synthetic binder and the mixture of fines prior to the introducing act. --
- -- 39. (previously presented) A method according to Claim 33 wherein the by-product tar is fed back mixed with another binder additive and combined with the mixture of coal fines and waste coke fines prior to the introducing act. --
- -- 40. (previously presented) A method according to Claim 32 wherein the discharging act comprises cooling the by-products and condensing tar to separate the tar from off-gas. --

-- 41. (currently amended) A method of producing coke from a mixture of non-prime coal fines and waste coke fines comprising the acts of:

introducing displacing without elutriation a mixture of non-elutriated low grade coal fines and another type of carbonaceous comprising non-elutriated waste coke fines as a feedstock influent into a pyrolyzer;

pyrolyzing the mixture in the pyrolyzer;

discharging segregated coke and pyrolytic by-products as effluents from the pyrolyzer. - -

-- 42. (previously presented) A method according to Claim 41 further comprising the acts of:

separating the pyrolytic by-products into tar and combustible off-gas;

combining the separated tar as a binder with the mixture of coal and coke fines in the mixture;

returning the combustible off-gas to the pyrolyzer as a source of fuel. - -

- - 43. (currently amended) A method according to Claim 41 wherein the introducing act comprises obtaining a mixture comprising non-elutriated waste coke fines and non-elutriated waste coal fines. -
- - 44. (previously presented) A method according to Claim 41 further comprising the act of crushing at least some of the coke and/or the coal, prior to the introducing act. -

- - 45. (previously presented) A method according to Claim 41 further comprising the act of forming the mixture into solid objects prior to the introducing act. -
- - 46. (previously presented) A method according to Claim 45 wherein the discharging act comprises discharging the coke from the pyrolyzer as solid objects. -
- - 47. (previously presented) A method according to Claim 42 wherein the combining act comprises combining the separated tar, a synthetic binder and the mixture of coal and coke fines prior to the introducing act. -
- - 48. (previously presented) A method according to Claim 42 wherein the separated tar is fed back to the coal and coke mixture prior to the introducing act. -
- - 49. (previously presented) A method according to Claim 42 wherein the separating act comprises cooling the by-products to condense tar to separate the tar from off-gas. -

-- 50. (currently amended) A method of producing coke from low grade coal and coke fines, comprising the acts of:

obtaining and mixing unwashed low grade coal fines and unwashed coke fines;

introducing displacing the unwashed mixture of lower grade coal fines and waste coke fines as an influent into a pyrolyzer without drying the fines;

pyrolyzing the unwashed mixture in the pyrolyzer;

discharging segregated coke, on the one hand, and pyrolytic by-products comprising combustible off-gas and tar on the other hand, as effluents from the pyrolyzer;

separating the pyrolytic by-products into segregated tar and combustible off-gas; adding the segregated tar as a binder to the coal and coke fines mixture; returning the segregated combustible off-gas to the pyrolyzer as a source of fuel. - -

- - 51. (previously presented) A method according to Claim 50 further comprising the act of crushing oversized unwashed waste coke and/or unwashed oversized low grade coal, to correctly size the fines. -
- - 52. (previously presented) A method according to Claim 50 further comprising the act of forming the mixture into solid objects to the introducing act. -
- - 53. (previously presented) A method according to Claim 52 wherein the discharging act comprises discharging the coke from the pyrolyzer as solid objects. -

- - 54. (previously presented) A method according to Claim 50 wherein the adding act comprises combining the separated tar, a synthetic binder and the mixture of coal and coke fines prior to the introducing act. -
- -- 55. (previously presented) A method according to Claim 50 wherein the separated tar is fed back to the mixture of coal and coke fines. --
- comprises 20-40% by weight of the coal and coke mixture. -
- - 57. (previously presented) A method according to Claim 50 wherein the coke fines comprise petroleum coke fines which comprise 40-70% by weight of the coal and coke mixture. -
- - 58. (previously presented) A method according to Claim 50 wherein the coke fines comprise coke breeze fines which comprise 5-10% by weight of the coal and coke mixture.
- - 59. (previously presented) A method according to Claim 50 wherein the pyrolyzing act comprises heating the introduced mixture to a temperature within the range of 800-1100°C at a rate within the range of 1500-2000°C/hour to lower coke volatility below 2%. -
- -- 60. (previously presented) A method according to Claim 50 wherein the separating act comprises cooling the by-products to about 300°C and condensing the tar to separate the tar from the off-gas. --

-- 61. (currently amended) A method of producing high quality coke from a mixture of low grade and/or waste carbonaceous materials at a much lower cost comprising the acts of:

absent elutriation, introducing displacing a mixture of low grade coal fines and waste coke fines as an influent into a pyrolyzer;

pyrolyzing the mixture of fines in the pyrolyzer;
discharging the coke, and pyrolytic by-products from the pyrolyzer. - -

comprise tar and combustible gas and further comprising the acts of:

condensing the tar;

using the tar as a binder for the mixture of coal and coke;
using the combustible off-gas as a source of fuel in the pyrolyzer. - -

-- 63. (currently amended) A method of producing coke from non-traditional carbonaceous materials comprising the acts of:

introducing displacing a mixture of unwashed waste coke fines and unwashed non-coking grade coal fines as an influent into a pyrolyzer absent washing and drying of the fines as they are introduced;

pyrolyzing the mixture in the pyrolyzer;

discharging the coke, and pyrolytic by-products comprising combustible off-gas and tar as effluents from the pyrolyzer. - -

- - 64. (previously presented) A method according to Claim 63 comprising the further acts of:

condensing the tar to separate the tar and off-gas;
using the tar as a binder for the mixture fines prior to the mixing act;
using the combustible off-gas as a source of fuel in the pyrolyzer. --

- - 65. (previously presented) A method according to Claim 64 wherein all condensed tar
 is utilized as binder and all combustible off-gas is used to fuel the pyrolyzer. -
 - - 66. (previously presented) A method according to Claim 64 wherein the condensed tar is the sole binder source and the combustible off-gas is the sole source of fuel for the pyrolyzer. -
 - - 67. (currently amended) A method of cost effectively producing high quality coke from a mixture of non-traditional carbonaceous materials comprising the acts of:

without washing or elutriation,

introducing displacing into a pyrolyzer a mixture comprising low grade coal fines and coke fines as salvage from prior production of coke without washing or elutriating the fines during the displacement;

pyrolyzing the mixture and obtaining segregated coke and by-products. - -

-- 68. (currently amended) A method of producing coke, comprising the acts of:
mixing a binder, low grade non-prime unwashed coal fines selected from the group consisting
of unwashed waste non-coking coal fines and unwashed non-coking coal fines and unwashed salvage
coke fines selected from the group consisting of unwashed waste petroleum fines, unwashed waste
char fines and unwashed waste coke breeze, without regard to a free swelling index value;

introducing displacing the mixture into a pyrolyzer without wetting or drying the displaced mixture;

pyrolyzing the mixture to derive coke, tar and combustible off-gas. --

- - 69. (previously presented) A method according to Claim 68 wherein the method is performed in a closed system and further comprising the acts of:

causing all of the tar to comprise the binder;

fueling the pyrolyzer with the combustible off-gas. - -

REMARKS

The Pending Claims

Claims 32 through 69 are pending.

The 35 USC § 112 Written Description Rejection

In the Office Action mailed 25 August 2003, the Examiner, for the first time and finally, rejected new claims 32-69 under 35 USC § 112, first paragraph, as failing to comply with the written description requirement because the terms "unwashed" and "non-elutriated" appear in the claims but are not expressly present in the specification.

The 35 USC § 112 Second Paragraph Rejection and Response Thereto

The Examiner's concern, forming the basis of her § 112, second paragraph, rejection has been fully addressed by cancellation of the phrases "on the one hand" and "on the other hand."

The Examiner Failed to Meet Her Prima Facie Burden

In ex parte proceedings, the Examiner bears the initial burden of presenting a prima facie case of unpatentability under 35 USC § 112. With respect to the written description requirement, the Examiner discharges his or her burden by presenting evidence, or reasons, why persons skilled in the art would not recognize in the disclosure a description of the invention set forth in the claims. However, if the specification contains a description of the invention, albeit not in the identical words, the Examiner must provide reasons why one of ordinary skill would not consider the description sufficient. This the Examiner failed to do. Only if the Examiner makes out a prima facie case does the burden shift to the Applicants to come forward with evidence showing that the invention as

presently claimed is adequately described to one of ordinary skill in the art. After the Applicants submit all of their evidence and arguments in response, the Examiner must determine patentability on the totality of the record, by a preponderance of the evidence, with due consideration to the persuasiveness of the Applicants' arguments.

Negative limitations in a claim, where the specification is silent as to such limitation may satisfy the written description requirement nevertheless. See <u>Ex parte Parks</u>, 300 USPQ2d 1234, 1236 (Bd. of Pat. Appeal and Intent; 1994) which states:

The examiner contends that the rejected claims lack adequate descriptive support because there is "no literal basis for the" claim limitation "in the absence of a catalyst." Clearly, the observation of a lack of literal support does not, in and of itself, establish a prima facie case for lack of adequate descriptive support under the first paragraph of 35 U.S.C. 112. In re Herschler, supra; In re Edwards, supra; In re Wertheim, supra.

* * * *

In the situation before us, it cannot be said that the originally-filed disclosure would not have conveyed to one having ordinary skill in the art that appellants had possession of the *concept* of conducting the decomposition step generating nitric acid in the <u>absence</u> of a catalyst. (Emphasized.)

The same analysis applies here.

Response to the Written Description Rejection

The first paragraph of 35 U.S.C. § 112 provides, in pertinent part, that "[t]he specification shall contain a written description of the invention."

Whether the written description requirement has been met is a question of fact. <u>Vas-Cath</u>, <u>Inc. v. Mahurkr</u>, 935 F.2d 1555, 19 USPQ2d 1111 (Fed. Cir. 1991); <u>Ralston Purina Co. v. Far-Mar-Co.</u>, 772 F.2d 1570, 227 USPQ 177 (Fed. Cir. 1985).

The Examiner erroneously concludes that simply because the terms "unwashed" and "nonelutriated" are not used per se in the specification, that, as a matter of fact, the apparatus and methodology which proceeds in the absence of washing and elutriating proves conclusively that the Inventors were not in possession of the claimed invention when the present application was filed.

The Applicants are not required to use any particular form of disclosure to describe the subject matter claimed, but rather, "the description must clearly allow one of ordinary skill in the art to recognize that he or she invented what is claimed." In re Gosteli, 872 F.2d 1008, 10 USPQ2d 1614 (Fed. Cir. 1989).

In essence, "the applicant must... convey with reasonable clarity to those skilled in the art that, as of the filing date sought, he or she was in possession of the invention." <u>Vas-Cath, Inc., supra</u>. Here, there can be no doubt that the Applicants' disclosed process and apparatus do not involve structure for or an act of washing the fines nor structure for or an act of elutriating to dry wet fines.

Exact correspondence between the language of the claims and words in the specification is not mandated by the written description requirement. See <u>In re Kaslow</u>, 217 USPQ 1089, 1096 (CAFC 1983) which states:

The test for determining compliance with the written description requirement is whether the disclosure of the application as originally filed reasonably conveys to the artisan that the inventor had possession at that time of the later claimed subject matter, rather than the presence or absence of literal support in the specification for the claimed language. In re Edwards, 558 F.2d 1349, 196 USPQ 465 (CCPA 1978); In re Herschler, 591 F.2d 693, 200 USPQ 711 (CCPA 1979). (Emphasis supplied.)

The attached Declaration of L. Douglas Smoot presents evidence showing conclusively that the Applicants have clearly disclosed apparatus and methodology which does not involve washing

or elutriating of the displaced fines to produce coke. Therefore, the Applicants' specification satisfies the written description requirement.

The Drawings Conclusively Show the Apparatus and Method Act Do Not Involve Process Washing or Process Elutriation

Realizing that the specification does not require extended recitations of what the invention is not, the drawings make it abundantly clear that the disclosed apparatus and methodology do not embrace washing and/or elutriation of fines being displaced to produce pyrolized coke. The drawings are conclusive of this point.

The drawings show no equipment for or method act of washing the fines being displaced during operation of the equipment. The drawings show no equipment for or method act by which fines should or could be displaced by elutriation. Thus, the drawings unequivocally show compliance with the written description requirement as to non-washing and non-elutriation.

As stated in Vas-Cath, Inc. v. Mahurkr, 19 USPQ2d 1111, 1117, 1118 (Fed. Cir. 1991):

... drawings alone may be sufficient to provide the "written description of the invention" required by § 112, first paragraph. Several earlier cases, though not specifically framing the issue in terms of compliance with the "written description" requirement, support this conclusion.

* * * *

These cases support our holding that, under proper circumstances, drawings alone may provide a "written description" of an invention as required by § 112. Whether the drawings are those of a design application or a utility application is not determinative. . . .

The Finality of the Rejection of 25 August 2003 is Premature and Should be Withdrawn

MPEP §§ 706.07, 706.07(c), 706.07(d), 706.07(e) and 716 controls when an Office Action should be made final and when the finality of an Office Action is to be withdrawn.

MPEP § 706.07 in pertinent part states:

Before final rejection is in order a clear issue should be developed between the examiner and applicant. To bring the prosecution to as speedy conclusion as possible and at the same time to deal justly by both the applicant and the public, the invention as disclosed and claimed should be thoroughly searched in the first action and the references fully applied ... Switching ... from one set of references to another by the examiner in rejecting in successive actions claims of substantially the same subject matter, will alike tend to defeat attaining the goal of reaching a clearly defined issue for an early termination; i.e., either an allowance of the case or a final rejection.

* * * *

... present practice does not sanction hasty and ill-considered final rejections. The applicant who is seeking to define his or her invention in claims that will give him or her the patent protection to which he or she is justly entitled should receive the cooperation of the examiner to that end, and not be prematurely cut off in the prosecution of his or her case.

* * * *

The examiner should never lose sight of the fact that in every case the applicant is entitled to a full and fair hearing, and that a clear issue between applicant and examiner should be developed, if possible, before appeal.

MPEP § 706.07(c) instructs as follows:

Any question as to prematureness of a final rejection should be raised, if at all, while the case is still pending before the primary examiner.

MPEP § 706.07(d) helpfully reads:

If, on request by applicant for reconsideration, the primary examiner finds the final rejection to have been premature, he or she should withdraw the finality of the rejection.

MPEP § 706.07(e) states:

The examiner may withdraw the rejection of finally rejected claims. If new facts or reasons are presented such as to convince the examiner that the previously rejected claims are in fact allowable.

MPEP § 716 states:

It is the responsibility of the primary examiner to personally review and decide whether affidavits or declarations submitted under 37 CFR 1.132 for the purpose of traversing grounds of rejection, are responsive to the rejection and present sufficient facts to overcome the rejection.

Similarly, MPEP § 716.01 states:

Evidence traversing rejections must be considered by the examiner whenever present. All entered affidavits, declarations, and other evidence traversing rejections are acknowledged and commented upon by the examiner in the next succeeding action. The extent of the commentary depends on the action taken by the examiner. Where an examiner holds that the evidence is sufficient to overcome the *prima facie* case, the comments should be consistent with the guidelines for statements of reasons for allowance. See MPEP § 1302.14. Where the evidence is insufficient to overcome the rejection, the examiner must specifically explain why the evidence is insufficient. General statements such as "the declaration lacks technical validity" or "the evidence is not commensurate with the scope of the claims" without an explanation supporting such findings are insufficient. (Emphasized.)

Please charge the fee associated with this Request to Withdraw Final, if any, to Deposit Account 06-1620 of the undersigned.

The Applicants should not be denied the right to fully respond to the new § 112 first paragraph written description rejection.

Entry of Amendment for Appeal Purposes

In the event the Examiner persists in her § 112 first paragraph position, she is requested to point out where washing of fines, displacement of wet fines, elutriation and drying of wet fines are disclosed in the present application.

It is requested that this Amendment be entered because it simplifies issues on appeal, should the Examiner not withdraw the present § 112 first paragraph rejection.

CONCLUSION

In the event it is found that the Examiner did meet her prima facie burden, the foregoing and the Declaration of L. Douglas Smoot clearly repute such by showing the Applicants were in possession of the presently claimed invention at the time this application was filed. The claims of this application are in condition for allowance and such action is courteously invited.

Respectfully submitted

ynn G. Foster

Attorney for Applicant

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of:

Craig N. Eatough, et al.

Docket:

8333

Serial No.: 09/954,603

Art Unit:

1764

Filed:

September 17, 2001

Examiner: Alexa A. Doroshenk

For:

CLEAN PRODUCTION OF COKE

CERTIFICATION-OF-FILING-BY-FACSIMILE-TRANSMISSION-

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

Sir:

I hereby certify the attached AMENDMENT was transmitted by facsimile to the aboveidentified Examiner at 703-872-9306 on the date indicated below.

Respectfully submitted

September 19, 2003

Attorney for Applicant

602 East 300 South Salt Lake City, UT 84102 Telephone: (801) 364-5633